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THE SEPARATION OF NEODYMIUM AND PRASEODYMIUM BY ION EXCHANGE II. THE EFFECT OF pH OF ELUENT ON THE ELUTION OF NEODYMIUM AND PRASEODYMIUM

A THESIS

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THE SEPARATION OF NEODYMIUM AND PRASEODYMIUM BY ION EXCHANGE II. THE EFFECT OF pH OF ELUENT ON THE ELUTION OF NEODYMIUM AND PRASEODYMIUM

Approved:

Thesis Advisor

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II. EFFECT OF pH OF ELUENT ON THE ELUTION OF NEODYMIUM AND PRASEODYMIUM.

A study has been made to determine the effect of pH of eluent, column loading, and flow rate on the elution of pure praseodymium and neodymium by ion exchange from a Dowex 50 resin bed using a five per cent citric acid solution of controlled pH as complexing agent. The effect of these variables has been interpreted in terms of the elution curves obtained when one variable was changed while maintaining the other variables constant.

The pH of the eluent was found to be the most important factor in determining the shape and position of the elution curves. By comparing the actual elution curves with the theoretical elution curves it is possible to predict the general shape of the adsorption isotherms. These comparisons indicated that the results could be explained in terms of an adsorption isotherm having a decreasing slope at pH 3.00, an increasing slope in the pH range 3.12 to 3.50, and a constant slope at some intermediate pH between 3.00 and 3.12.

Increasing the column loading of Nd_2O_3 resulted in an increase in the band width and an increase in the peak concentration. At pH 3.20, the peak concentration was directly proportional to the column loading over the range of column loadings from 3.60 to 13.0 grams of Nd_2O_3.

The flow rate experiments at a column loading of 13.0 grams of Nd_2O_3 indicated that the greater flow rate required less volume of eluent to produce break-through, increased the width of the elution band, and decreased the peak concentration with no appreciable change in the position of the peak. Similar results were obtained at a column loading of 3.60 grams of Nd_2O_3, but in the latter case the peak of the elution curve was reached with less volume of eluent at the greater flow rate.

Flow rate experiments undertaken by Spedding[1] indicated that increasing the flow rate increased the volume of eluent necessary to produce break-through, that is, the entire elution band was shifted together with a widening of the elution band and a lowering of the peak. Spedding's experiments were carried out on Amberlite IR-100 resin beds using 0.5 per cent citrate solutions.

The discrepancy (in the effect of flow rate on the position of the break-through) between the results of the flow rate experiments carried out in this thesis and those carried out by Spedding is not readily explained. Further experimental work is necessary to fully understand the non-equilibrium elution process.

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[1]Spedding, F.H., Fulmer, E.I., Butler, T.A., and Powell, J.E., J. Am. Chem. Soc., 72, 2349 (1950).

THE SEPARATION OF NEODYMIUM AND PRASEODYMIUM BY ION EXCHANGE

II. EFFECT OF pH OF ELUENT OF THE ELUTION OF NEODYMIUM AND PRASEODYMIUM

I

INTRODUCTION

In the past two years a series of experiments has been carried out in the low temperature laboratory of the State Engineering Experiment Station on the separation of praseodymium and neodymium by ion exchange. The initial studies, undertaken by W. T. Ziegler, et al.,[1] consisted of about twenty-four column runs. These runs were made on Dowex 50 columns using a five per cent citric acid solution of controlled pH as complexing agent.[2] The main objective of these experiments was the production of moderate quantities (10-50 grams) of praseodymium and neodymium oxides having a purity of 99.5 per cent or better, employing commercially available rare earth concentrates as starting material.

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[1] Blomeke, J.O., Roberts, J.T., Jr., and Ziegler, W.T., Paper presented before Georgia section - American Chemical Society, Meeting in Miniature, November 17, 1949, together with unpublished work.

[2] The complexing agent referred to throughout this paper as five per cent citric acid was prepared by dissolving 900 grams of citric acid monohydrate and 18 grams of phenol in 18 liter of distilled water and adding concentrated ammonium hydroxide to give the desired pH.

The greater part of this work was carried out on Dowex 50 columns having a diameter of 4.7 cm and a length of approximately 75 cm. The selection of the operating conditions for the work mentioned above was based primarily on literature presented in an ion exchange symposium.[3]

These initial studies were followed by those of Johnson[4] which were carried out during the period January - June (1950). The studies undertaken by Johnson were made on Dowex 50 resin beds having a diameter of 4.7 cm and a length of 120 cm. The purpose of these studies was to determine the effect of column loading and pH of eluent on the separation of a mixture of rare earth oxides using a 120 cm resin bed and to compare the results of these experiments with those obtained on the shorter (75 cm) columns.

The present thesis has as its objective the study of the effect of pH of eluent, column loading, and flow rate on the elution of pure neodymium and praseodymium. The experimental work consisted of sixteen column runs which were carried out during the period March - August (1950). All runs were made using Dowex 50 in the ammonium form as the exchanging medium. The resin beds were 4.7 cm in diameter and had a length of 73.5 ± 1 cm. The complexing agent employed was five per cent citric acid.

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[3] This symposium was published in the J. Am. Chem. Soc., 69, 2769-2881 (1947).

[4] Johnson, W.N., Master's Thesis in Chemical Engineering, Georgia Institute of Technology, (1950).

In order to determine the effect of the pH of the eluent on the elution of praseodymium and neodymium, two series of runs were made in which the pH of the eluent was varied while the column loading and flow rate were held constant. The column loading, pH of eluent, and flow rate for each of these runs are given in Table I and Table II.

TABLE I

Effect of pH on the Elution of Praseodymium for Column Loading of 13.0 Grams of Pr_6O_{11}

Run Number	pH of Eluent	Column Loading gm Pr_6O_{11}	Average Flow Rate ml/min
38	3.00	13.041	20.0
36	3.12	13.033	19.4
42	3.20	13.017	19.4
49	3.50	12.994	18.9

TABLE II

Effect of pH on the Elution of Neodymium for Column Loading of 13.0 Grams of Nd_2O_3

Run Number	pH of Eluent	Column Loading gm Nd_2O_3	Average Flow Rate ml/min
37	3.00	13.052	20.6
45	3.11	13.001	20.0
43	3.20	13.024	20.2
48	3.50	13.161	19.4

To determine the effect of column loading on the elution of neodymium a series of runs was made using smaller column loadings and maintaining the other variables at essentially the same values as those in Table II. The column loading, pH of eluent, and average flow rate for each of these runs are given in Table III.

TABLE III

Effect of pH on the Elution of Neodymium

Run Number	pH of Eluent	Column Loading gm Nd_2O_3	Average Flow Rate ml/min
46	3.11	3.602	10.0
44	3.20	3.602	20.4
32	3.20	10.000	18.6
50	3.50	3.600	19.3

The effect of flow rate on the elution of neodymium was determined by comparing a series of runs in which the flow rate was varied while maintaining the column loading and the pH of the eluent constant. Two series of flow rate experiments were made, one with a column loading of approximately 13.0 gm of Nd_2O_3 and another with a column loading of approximately 3.60 gm of Nd_2O_3. The column loading, pH of eluent, and flow rate used in these runs are given in Table IV.

TABLE IV

Effect of Flow Rate on the Elution of Neodymium

Run Number	pH of Eluent	Column Loading gm Nd_2O_3	Average Flow Rate ml/min
40	3.11	13.122	10.3
45	3.11	13.001	20.0
41	3.11	13.002	31.3
46	3.11	3.602	10.0
47	3.11	3.602	32.0

CHAPTER II

THEORY OF COLUMN OPERATION

In order to explain the theory of ion exchange, the theory of chromatography will be presented and it will then be shown that the results obtained for chromatography can be applied to ion exchange. Wilson[5] points out that the so-called chromatographic adsorption method of analysis was originated by the Russian botanist M. T. Tswett. Wilson describes Tswett's discovery in the following manner:

> Tswett discovered that if a solution containing a mixture of colored solutes is allowed to run through a vertical glass tube filled with a suitable powdered adsorbing material, the material adsorbed in the column appears as a series of colored bands, indicating that a partial separation of the components of the solution has taken place. This series of colored bands is known as a chromatogram. The separation can be completed by a procedure known as development of the chromatogram: there is poured through the column a suitable solvent which washes the colored bands down the tube at different rates, the lowest-lying band moving the fastest. If this process is carried out in a sufficiently long tube with the use of a sufficiently large volume of solvent it is possible to effect complete separation of the original components of the solution into a series of discrete bands separated by clear spaces of adsorbent.

Wilson has developed a theory of chromatography based on the assumption that equilibrium between solution and adsorbent is instantaneously established and the effects of diffusion can be neglected. DeVault[6] has extended this theory and points out certain important results given by a treatment similar to Wilson's but not discussed by

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[5] Wilson, J.N., J. Am. Chem. Soc., 62, 1583 (1940).

[6] DeVault, D., J. Am. Chem. Soc., 65, 532 (1943).

him. The theory is based on the solution of the differential equation for the transfer of solute between the solution and the adsorbent. The differential equation will be developed below for the case of a single solute. The derivation of the differential equation and the nomenclature used follow that presented by DeVault.

α = pore volume per unit length of column

M = amount of adsorbing material per unit length of column

V = volume of solution at any time that has been poured into the column since the initial time

x = distance from any point on the column to the top of the column

c = c(V,x) = amount of solute in solution per unit volume of solution, as a function of time and position in the column

Q = Q(V,x) = amount of solute adsorbed per unit length, as a function of time and position in the column

c_o = concentration of solution entering the column

f = f(c) = adsorption isotherm of the solute on the adsorbent such that Q = Mf

Consider a cross sectional layer of the column of thickness dx. Let a volume dV of solution enter this section. The difference between the amount of solute entering and leaving the section will be $(\frac{\partial c}{\partial x})dx\ dV$. The amount of solute in solution in the section increases by the amount $dx(\frac{\partial c}{\partial V}\ dV)$. The amount of solute adsorbed in the section increases by the amount $dx(\frac{\partial Q}{\partial V}\ dV)$. Combining these expressions to form an equation for the conservation of matter and cancelling dx and dV we obtain

$$\frac{\partial c}{\partial x} + \alpha\frac{\partial c}{\partial V} + \frac{\partial Q}{\partial V} = 0 \qquad (1)$$

This may also be written

$$\frac{\partial c}{\partial x} + \left[\alpha + Mf'(c)\right] \frac{\partial c}{\partial V} = 0 \qquad (2)$$

where f' is the first derivative of f. DeVault has shown that the general solution of the above differential equation is

$$c = \varphi(V - x\left[\alpha + Mf'(c)\right]) \qquad (3)$$

where φ is any function determined partly by the initial conditions.

The details of the mathematics will not be presented herein but the results of DeVault will be presented in terms of the curves in Figure 1 for three typical adsorption isotherms. In Figure 1, the curves in the first row represent three typical adsorption isotherms. The results in column B represent the special case of a linear adsorption isotherm, B1, and this special case was first treated by Wilson. The curves in the second row represent the assumed distribution of solute in the solution in the column before any of the solvent has started through the column. Curves representing the concentration of solute on the resin would have a shape similar to those in the second row since Q, the concentration of solute on the resin is related to c, the concentration of solute in the solution, by the relation $Q = Mf(c)$. The curves in the third and fourth row represent the distribution of solute in the solution in the column after successive volumes of solvent have passed through the column. The curves in the fifth row represent the theoretical elution curves. An elution curve is a plot of the concentration of solute in the effluent versus the volume of effluent which has passed a point x_L in the column, where x_L is at the bottom of the column.

Figure 1

Band Shapes in Equilibrium Elution

Legend

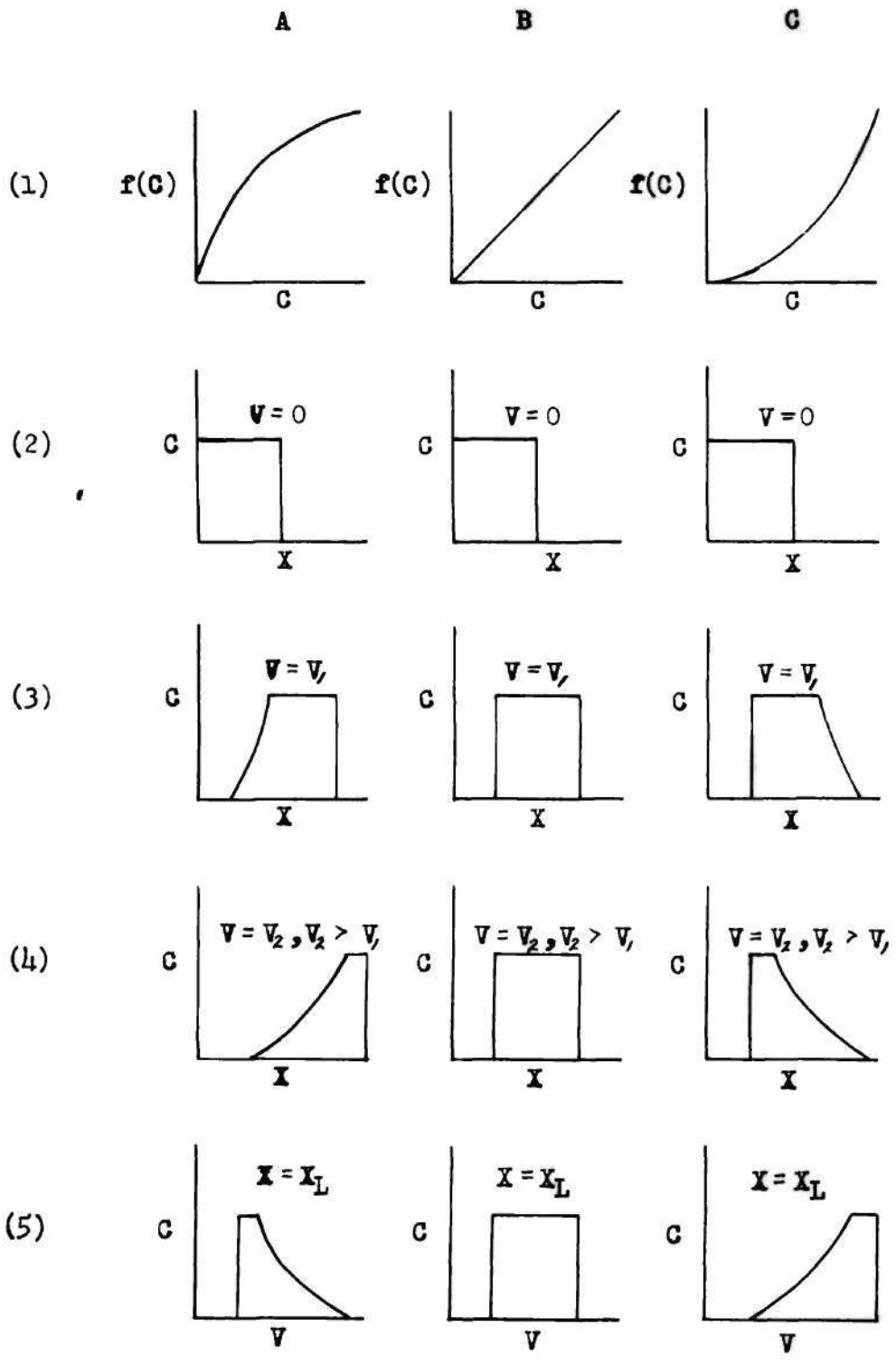
$f = f(c)$ = adsorption isotherm of the solute on the adsorbent

$c = c(V,x)$ = amount of solute in solution as a function of time and position in the column

x = distance from top of adsorbent bed

x_L = bottom of column

V = volume of solution at any time that has been poured into the column since the initial time


A
B
C
(1)
f(C)
C
f(C)
C
f(C)
C
(2)
V = 0
C
X
V = 0
C
X
V = 0
C
X
(3)
V = V,
C
X
V = V,
C
X
V = V,
C
X
(4)
V = V₂ , V₂ > V,
C
X
V = V₂ , V₂ > V,
C
X
V = V₂ , V₂ > V,
C
X
(5)
X = X_L
C
V
X = X_L
C
V
X = X_L
C
V

On examining the band shapes in row 3 and 4 for the adsorption isotherms A1 and C1 it is noted that as the bands move down the column the flat region on the top of the bands becomes narrower. As the bands continue down the column the flat region will eventually disappear and at this point the maximum heighth of the band will begin to drop. However, for adsorption isotherms having the shape B1, movement of the bands down the column will not result in a change in the shape of the band. In each of the cases shown a certain minimum volume of solvent must pass through the column before the band shapes in row 2 will change to those shown in row 3. It should also be mentioned that while we have assumed a certain initial distribution of solute along the column, DeVault's treatment is quite general and can be applied to cases where other initial distributions and adsorption isotherms are encountered.

We will now consider the reactions which take place on an ion exchange column, arbitrarily assuming the behavior of a trivalent neodymium ion to be typical for any trivalent rare earth ion. These reactions consist of an ion exchange reaction and a complexing reaction.

$$3NH_4^+ + NdR \rightleftharpoons Nd^{+++} + 3NH_4R \quad \text{(ion exchange)} \qquad (4)$$

$$Nd^{+++} + 3H_2Cit^- \rightleftharpoons Nd(H_2Cit^-)_3 \quad \text{(complexing)} \qquad (5)$$

Ketelle and Boyd[7] have applied the mass law to the above equilibria and

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[7] Ketelle, B.H., and Boyd, G. E., J. Am. Chem. Soc., 69, 2800 (1947).

obtained the following expression for the equilibrium distribution coefficient, K_d,

$$K_d = \frac{NdR}{Nd(H_2Cit^-)_3} = \frac{K^{exchange}\ K^{complex}}{K_1^3} \frac{(NH_4R)}{(NH_4^+)} \frac{(H^+)^3}{(H_3Cit)^3} \qquad (6)$$

where K_1 is the first dissociation constant for citric acid. In the pH range below 3.5 the H_2Cit^- ion is the predominant citrate ion.[8] In the above expression NdR and $Nd(H_2Cit)_3$ represent the concentration of neodymium in the resin phase and the solution phase, respectively. R represents the anionic part of the resin phase and the concentrations are expressed as thermodynamic activities. Ketelle and Boyd have made some important qualitative deductions from the above expression for the equilibrium distribution coefficient. These are (1) increasing the pH (accomplished in practice by adding ammonium hydroxide) causes the hydrogen ion concentration to decrease and the ammonium ion to increase so that the magnitude of the distribution coefficient will decrease sharply. The decrease in the concentration of the undissociated citric acid will tend to offset this change but only to a slight degree. (2) Increasing the citric acid concentration also causes a decrease in the magnitude of K_d. Therefore, the value of K_d, which represents the affinity of the rare earth for the resin, can be varied by changing the pH or the citric acid concentration.

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[8] Tompkins, E.R., and Mayer, S. W., J. Am. Chem. Soc., 69, 2859 (1947).

The role of the citrate complex in a separation process can be shown by its effect on a separation factor, α, defined by Ketelle and Boyd[9] as the ratio of the distribution coefficients of two neighboring rare earths. Ketelle and Boyd have demonstrated this effect for the pair of rare earth ions, Ce^{+++} and Y^{+++}. From the definition of the separation factor, α, and Equation (6),

$$\alpha = \frac{K_d^{Ce}}{K_d^{Y}} = \frac{K^{Ce}{}_{exchange}}{K^{Y}{}_{exchange}} \quad \frac{K^{Ce}{}_{complex}}{K^{Y}{}_{complex}} \qquad (7)$$

Ketelle and Boyd report that a value of 1.55 has been observed for the first ratio on the right hand side of Equation (7) and that the second ratio is known to possess a value of approximately 2.9. The introduction of the citrate has thus induced a considerable change in the relative adsorbabilities of the two rare earths.

Since the differential equation developed for the transfer of solute between the solution phase and the solid phase for chromatography was independent of the mechanism governing the transfer, the differential equation is also applicable to the case of ion exchange. The adsorption isotherms, that is, the distribution of rare earth ion between the resin phase and the solution phase would now be governed by the factors in equation (6).

Bauman and Eichhorn[10] have plotted isotherms based on the mass action law and obtained curves having the same general shape as those

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[9] Ketelle, B.H., and Boyd, G.E., J. Am. Chem. Soc., 69, 2800(1947).

[10] Bauman, W.C., and Eichhorn, J., J.Am. Chem. Soc., 69, 2859(1947).

in Figure 1, In checking these equations in the ammonium - hydrogen exchange for Dowex 50 at various salt concentrations Bauman and Eichhorn found that the experimental data are in good agreement with the mass action law at concentrations up to 1.0 N. Walter [11] has solved the differential equation of mass transfer using an adsorption isotherm based on the mass action law for the case of monovalent-monovalent exchange and obtained elution curves which have the same general shape as those shown in Figure 1.

Tompkins and Mayer[12] have determined experimental adsorption isotherms for the exchange of certain rare earth ions in 0.23 M citrate solutions (approximately 5 per cent citric acid solution) with Dowex 50. In their experiments it was reported that the rare earth concentration does not effect K_d in concentrations up to about 6×10^{-3} M, but at rare earth concentrations above 6×10^{-3} M K_d was found to decrease with increasing concentration of rare earth. In other words, the adsorption isotherms would be linear at concentrations below 6×10^{-3} M and would have a decreasing slope at concentrations above 6×10^{-3} M.

In practice the elution process is carried out under non-equilibrium conditions. The theory of the non-equilibrium elution process has not been very completely developed although rather extensive experimental studies have been made by several investigators. A brief survey of the theoretical and experimental studies which have been made to

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[11] Walter, J.E., J. Chem. Phys., 13, 229 (1945).

[12] Tompkins, E.R., and Mayer, S.W., J. Am. Chem. Soc., 69, 2859 (1947).

determine the mechanism of the rate controlling process is presented by Nachod.[13]

Although a theoretical discussion will not be included herein, we will point out the kinetic processes which must be considered. Boyd, et al.,[14] point out that the kinetic processes involved are (1) diffusion through a liquid film at the periphery of the particle (2) diffusion in and through the adsorbent particles (3) chemical exchange in the interior of the particle. An experimental study by Boyd, et al., using trace concentrations of alkali metal cations and Amberlite IR-1 resin revealed that in their experiments diffusion was the rate controlling process. Other conclusions drawn by Boyd, et al., were that the primary factors determining the nature of the rate controlling mechanism were the distribution constant K_d and the particle radius r_o. Large values of K_d and/or small values of r_o favor a rate determined by film diffusion if the temperature and flow rate are kept constant.

Boyd, et al.,[15] have discussed the qualitative effects of non-equilibrium column operation for linear adsorption isotherms in terms of the curves in Figure 2. In Figure 2 it will be noted that the leading and trailing edges are diffuse in contrast to the sharp edges found in curves B3, B4, and B5 in Figure 1.

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[13] Nachod, F.C., Ion Exchange Theory and Application, New York: Academic Press, 29-43 (1949).

[14] Boyd, G.E., Adamson, A.W., and Myers, L.S. Jr., J. Am. Chem. Soc., 69, 2836 (1947).

[15] Boyd, G.E., Myers, L.S. Jr., and Adamson, A.W., J. Am. Chem. Soc., 69, 2849 (1947).

Figure 2

Band Shapes in Non-equilibrium Elution

for Linear Adsorption Isotherms

Legend

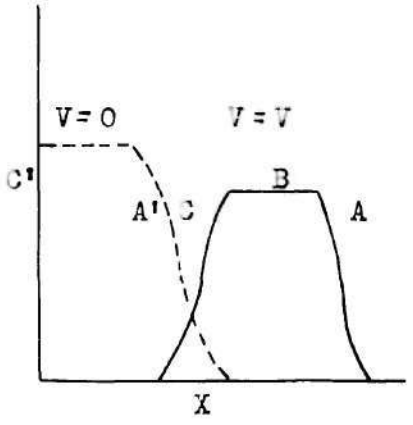
C' = concentration of cation on the resin

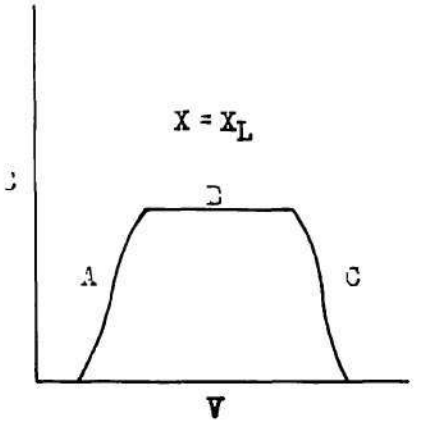
x = distance from the top of the resin bed

c = concentration of cation in the effluent

V = volume of effluent which has passed through the column

x_L = bottom of column


V = 0
V = V
C'
A'
C
B
A
X


X = X_L
B
A
C
V

Two different shapes are possible for the elution bands in Figure 2. The curves as shown with the flat middle region, B, are formed under certain operating conditions. Under somewhat different operating conditions it is possible to form a band in which the branches A and C will overlap and the flat middle region, B, will not be exhibited.

Although the curves in Figure 2 represent the case of linear adsorption isotherms, the results can be extended to the other isotherms shown in Figure 1. For the case of non-linear adsorption isotherms, the sharp edges of the bands would become diffuse and the edges which are already diffuse would become more diffuse under non-equilibrium operating conditions.

CHAPTER III

EXPERIMENTAL

Apparatus

The column (Figure 3) used for these experiments consisted of a cylindrical glass tube, A, having an inside diameter of 4.7 cm and a length of 150 cm. A coarse porosity fritted glass plate, B, was sealed into one end of the tube to support the resin bed, C. Two 20 liter carboys, E, served as containers for the storage of eluent and a 200 cc glass bottle, F, served as a constant head regulator. The flow rate was adjusted by means of the screw clamp, D. A turntable and timing device provided for the automatic collection of the individual fractions in the erlenmeyer flasks, J. A layer of liquid, G, was maintained above the resin at all times to prevent entrainment of air in the resin bed.

A Beckman model G glass electrode pH meter, accurate to $\pm$0.02 pH units, was used to measure the pH of the adsorbate solution, eluent, and individual fractions. To insure reproducible pH measurements the meter was first set against a standard buffer of pH 7.00 and then checked against a standard buffer of pH 4.00. The meter was found to check the pH 4.00 buffer to within $\pm$0.02 pH units. The above standardizing procedure was employed before and after the meter was used to determine a group of measurements.

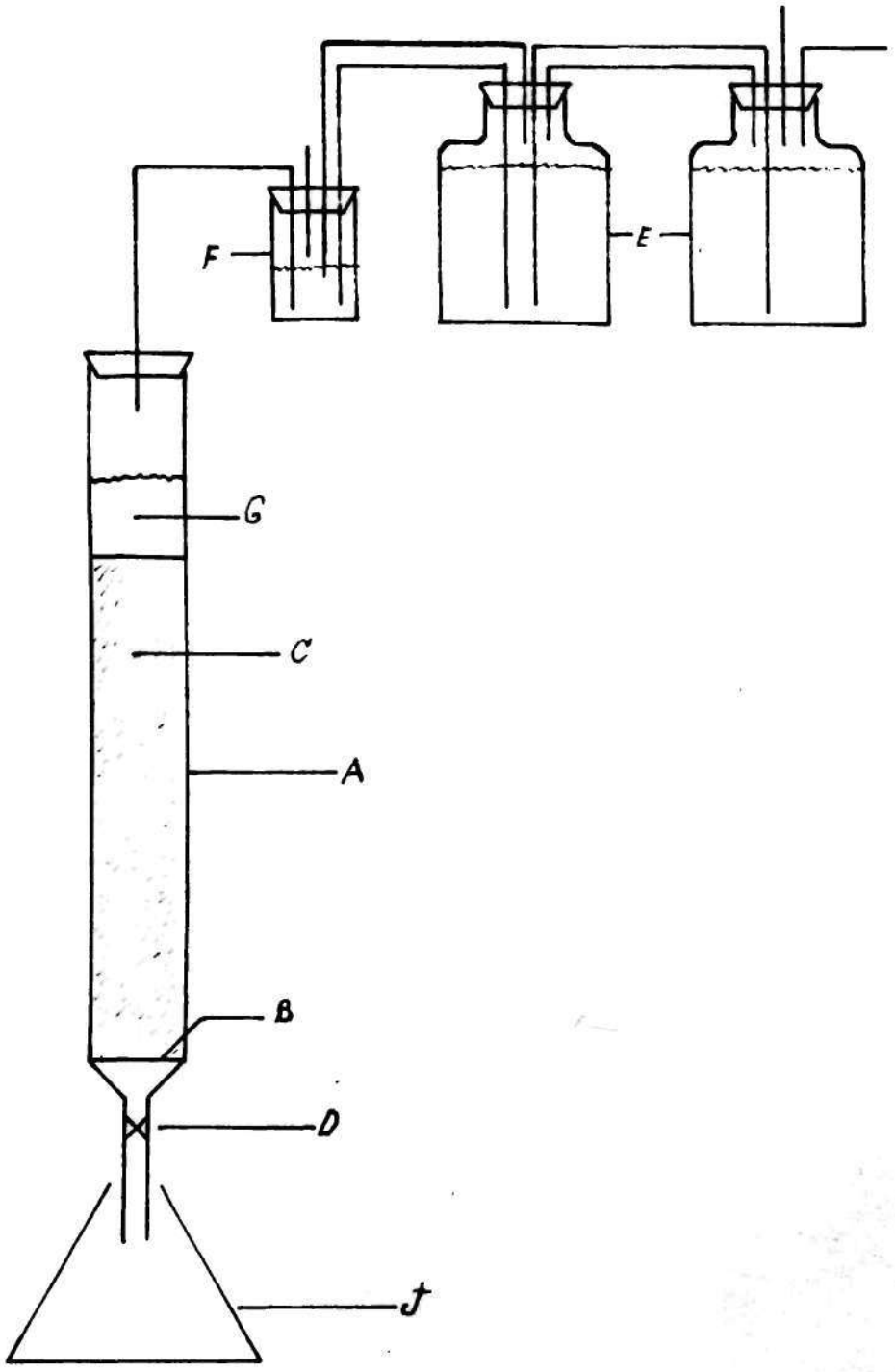
Materials

Dowex 50 resin was used for all experiments in this thesis. The resin was purchased from the National Aluminate Company and is sold un-

Figure 3

Schematic Diagram of Column Used in the

Elution of Rare Earths


F
E
G
C
A
B
D
J

der the trade name, Nalcite HCR, and is specified as being 60-100 mesh. The resin, as received, was in the sodium form and was given the following preliminary treatment. The resin was washed with distilled water until the supernatant liquid became clear; it was then washed into the column and converted to the hydrogen form by passing 4-5 liters of 2M HCl through the column. The resin was then converted to the ammonium form by passing five per cent citric acid, adjusted to a pH of 3.95 (approximately 0.43 M in NH_4^+) by the addition of concentrated ammonium hydroxide, through the column until equilibrium was attained, i.e., the pH of the effluent reached 3.95. About 16-18 liters of ammonium citrate solution were used in the latter process. The volume of solution used in the above conversion processes represents an excess of about 100 per cent over the stoichiometric quantity. The resin was then washed out of the column, filtered to remove the excess water, and stored in the wet form. When the present work was begun, a weighed amount of wet resin was washed into the column to form the resin bed. Previous measurements carried out by Roberts[16] on the wet resin indicated that approximately 1100 grams of wet resin would form a resin bed of about 73.5 cm on a column having an inside diameter of 4.7 cm. Other measurements by Roberts showed that 1.57 grams of wet resin were equivalent to 1 gram of oven dried resin (120° C). The column used in these experiments contained 1100 grams of wet resin or 700 grams of dry resin. This weight of resin is equal to approximately 3.8 equivalents.

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[16] Roberts, J.T., Jr., unpublished work, Georgia Institute of Technology (1950).

Previous experiments in this laboratory indicated that five per cent citric acid solution adjusted to a pH of about 4.00 would readily remove lanthanum and other rare earths from the resin and convert the resin to the ammonium form. In order to have the resin in the same condition for each run it was decided to equilibrate the resin with five per cent citric acid solution of pH 3.95 before each run.

The praseodymium oxide used in the experiments was obtained from separation processes carried out earlier in this laboratory. The neodymium oxide used in these experiments came from two sources, laboratory stock from previous runs and a supply of neodymium oxide which was purchased from Research Chemicals Inc., 831 North Lake Street, Burbank, California. This latter oxide, designated as Lot No. 239, was described by the manufactures as 99 per cent neodymium oxide. The praseodymium and neodymium taken from laboratory stock was believed to have a purity of about 99 per cent.

A rough check on the purity of the Nd_2O_3 is possible by examining the colors of the freshly ignited oxide obtained in each fraction. Pure Pr_6O_{11} is black and pure Nd_2O_3 is pale blue. Previous experiments in this laboratory have shown that a small amount of Pr_6O_{11} mixed with pure Nd_2O_3 gives a mixture having a tan color. The detection of any praseodymium is enhanced by the elution process which tends to separate the neodymium from the praseodymium. Any praseodymium present would thus tend to be concentrated in the last fractions removed from the column. Estimation of the purity of the oxide in this manner verifies the purities given above. The color of the oxide in each fraction for each

column run is recorded in Appendix I.

The five per cent citric acid solution used as eluent was prepared by dissolving 900 grams of citric acid monohydrate (technical grade, Pfizer) in 18 liters of distilled water. Eighteen grams of phenol (1 gram per liter) were added to prevent mold formation.[17] The pH was adjusted to the desired value by the addition of concentrated ammonium hydroxide. A sample of each batch of eluent was checked for the presence of any interfering ions by the addition of saturated oxalic acid solution.

Column Operation

The rare earth was placed on the column in the form of the rare earth chloride solution which was prepared by dissolving the freshly ignited rare earth oxide in a slight excess (5 per cent) of 1N HCl and neutralizing with ammonium hydroxide to give the solution a pH in the range 2-2.5. The neutralized solution was then made up to the desired volume by adding distilled water. The final volume of the loading solution, designated as the adsorbate solution, was 500 ml for the 13.0 gm column loadings and 350 ml for the 3.6 gm column loadings.[18] The flow rate during the loading period varied from 10 to 30 ml/min. After loading the column was washed with 500 ml of distilled water. After

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[17] Spedding, F.H., Fulmer, E.I., Butler, T.A., Gladrow, E.M., Gobush, M., Porter, PIE., Powell, J.E., and Wright, J.M., J. Am. Chem. Soc., 69, 2812 (1947).

[18] The term "column loading" as used throughout this paper means the weight of the freshly ignited oxide which has been adsorbed on the column.

washing the column with distilled water, eluent was started through the column and the flow rate adjusted by means of the screw clamp in the outlet line.

The average flow rate during each fraction could be readily determined since the collection periods for each fraction were fixed by the setting of the timing device and the volume of each fraction was measured. The average deviation and the maximum deviation are shown in Table V for each run as percentages of the overall average flow rate. Since Table V does not give a very complete picture of the variation of flow rate, a typical flow rate curve has been included in Figure 4.

The effluent was collected in erlenmeyer flasks; the size of the individual fractions collected depended upon the estimated concentration of rare earth in the effluent. The volume of the individual fractions varied from 400 to 1300 ml.

The progress of the elution process could be followed by periodic measurements of the pH of the individual fractions, for, as the concentration of the rare earth in the effluent increases the pH of the effluent decreases.

Each fraction was tested for the presence of rare earth by adding 20-60 ml of saturated oxalic acid solution, allowed to stand overnight, and then tested for complete precipitation. When the effluent fractions no longer yielded precipitates, eluent of pH 3.95 was started through the column. These additional fractions were also tested for the presence of rare earth ions. Elution was stopped when the pH of the effluent reached 3.95. The column was then washed with 4-5

TABLE V

Variation of Flow Rate

Run Number	Average Flow Rate ml/min	Average Deviation %	Maximum Deviation %
32	18.6	9.7	22.0
36	19.4	5.4	16.8
37	20.6	2.2	8.4
38	20.0	3.4	34.7
39	20.4	1.0	2.8
40	10.3	1.8	6.2
41	31.3	2.7	5.4
42	19.4	6.1	12.3
43	20.2	.9	4.0
44	20.4	2.4	11.0
45	20.0	1.9	7.5
46	10.0	3.9	16.7
47	32.0	2.7	6.2
48	19.4	2.0	8.8
49	18.9	2.8	9.7
50	19.3	3.5	13.0

Figure 4

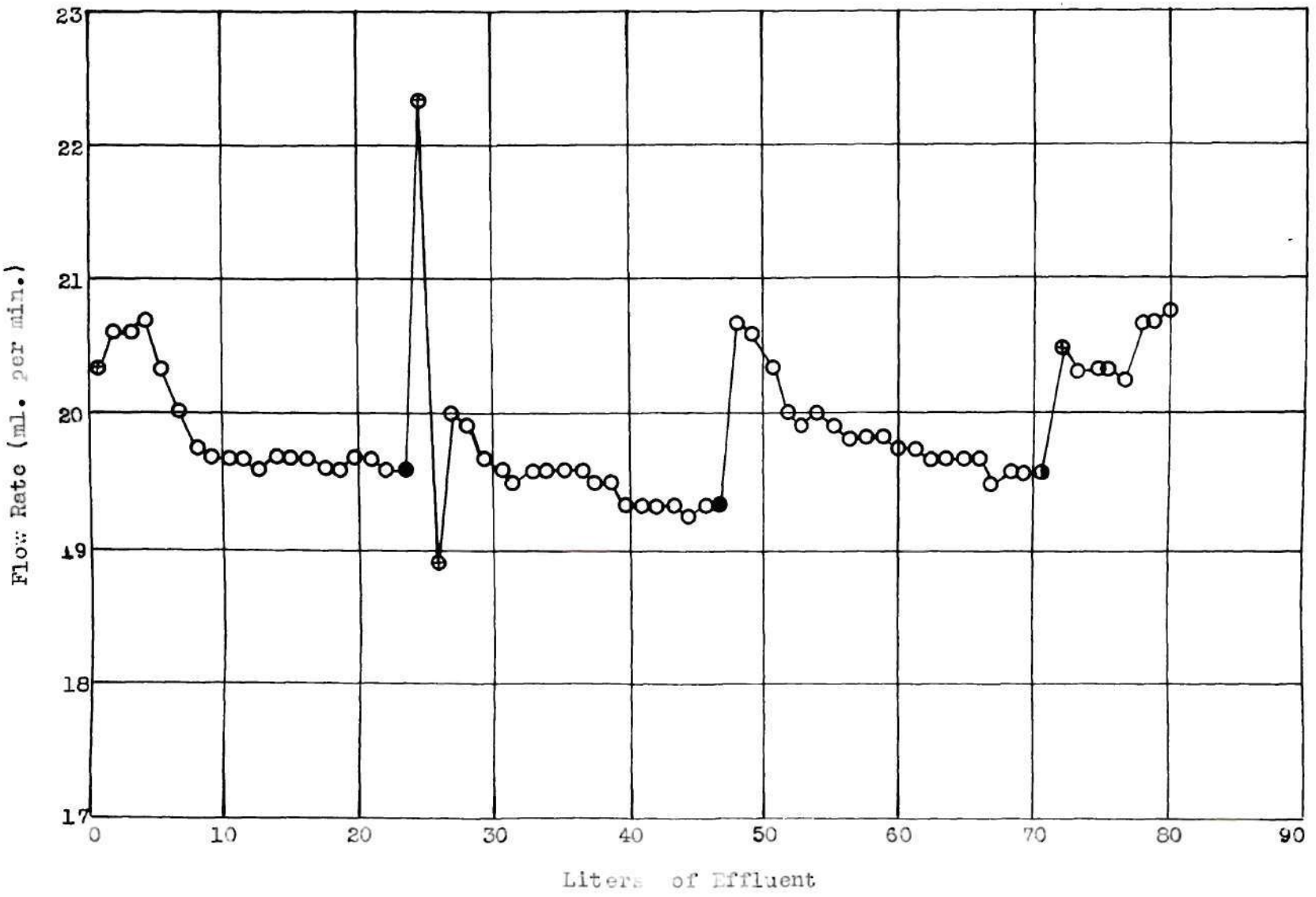
Typical Flow Rate Curve (Run 38)

Legend

⊕ Adjusted flow rate during fraction

● New batch of eluent added to overhead storage after this fraction

◐ Column operation stopped for several hours after this fraction


Flow Rate (ml. per min.)
23
22
21
20
19
18
17
0
10
20
30
40
50
60
70
80
90
Liters of Effluent

liters of distilled water.

The fractions containing precipitates were filtered through No. 41 Whatman filter paper. The precipitates were then washed with distilled water, placed in weighed porecelain crucibles, and dried in an oven at 120° C. The filter paper was ashed over a gas flame and the precipitates ignited for at least eight hours in a muffle furnace at 850° C. The crucibles were then removed from the furnace, allowed to cool for 5-10 minutes and placed in a desiccator while still warm. The crucibles were weighed immediately after reaching room temperature.

A summary of the experimental data for each column run is presented in Appendix I.

CHAPTER IV

DISCUSSION OF RESULTS

The purpose of this thesis was to determine the effect of pH of eluent, column loading, and flow rate on the elution of praseodymium and neodymium by ion exchange from a Dowex 50 resin bed. The effect of these variables can best be determined by comparing the elution curves obtained when one variable is changed while maintaining the other variables constant. An elution curve, as has been previously described, is a plot of the concentration of the rare earth in the effluent as a function of the volume of effluent which has passed through the column.

Effect of pH of Eluent

Previous studies made in this laboratory[19] and experiments by other investigators[20] have shown that the pH of the eluent has the greatest effect on the elution process. The effect of pH of eluent on the elution of pure praseodymium and neodymium is illustrated by the elution curves in Figures 5, 6, and 7. At pH 3.00 the elution curves are longer on the trailing edge than on the leading edge, but at pH 3.12, 3.20, and 3.50 the elution curves are longer on the leading edge than on the trailing edge. It would seem likely that at some intermediate pH between 3.00 and 3.12 the elution curves would become approximately symmetrical.

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[19] Ziegler, W.T., et al., unpublished work, Georgia Institute of Technology (1948-1949).

[20] J. Am. Chem. Soc., 69, 2769-2881 (1947).

Figure 5

Effect of pH on the Elution of Praseodymium for

Column Loading of 13.0 grams of Pr_6O_{11}

Curve Designation	Run Number	pH of Eluent	Column Loading gm Pr_6O_{11}	Average Flow Rate ml/min
⊕—⊕—⊕—⊕	38	3.00	13.048	20.0
◒—◒—◒—◒	36	3.12	13.033	19.4
○—○—○—○	42	3.20	13.017	19.4
●—●—●—●	49	3.50	12.994	18.9

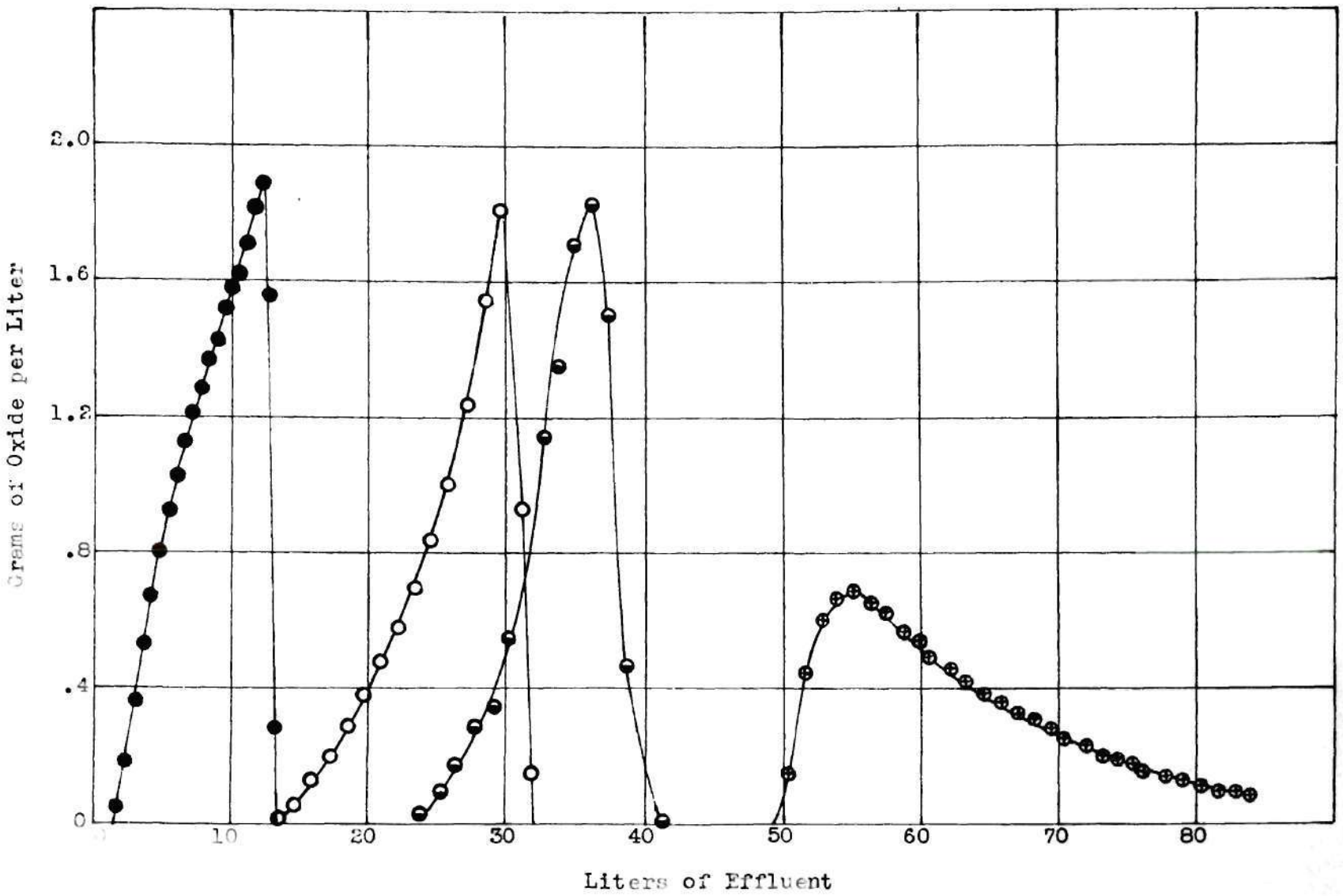
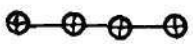
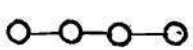

2.0
1.6
1.2
.8
.4
0
10
20
30
40
50
60
70
80
Grams of Oxide per Liter
Liters of Effluent

Figure 6

Effect of pH on the Elution of Neodymium for

Column Loading of 13.0 grams of Nd_2O_3

Curve Designation	Run Number	pH of Eluent	Column Loading gm Nd_2O_3	Average Flow Rate ml/min
	37	3.00	13.052	20.6
	45	3.11	13.001	20.0
	43	3.20	13.024	20.2
	48	3.50	13.161	19.4

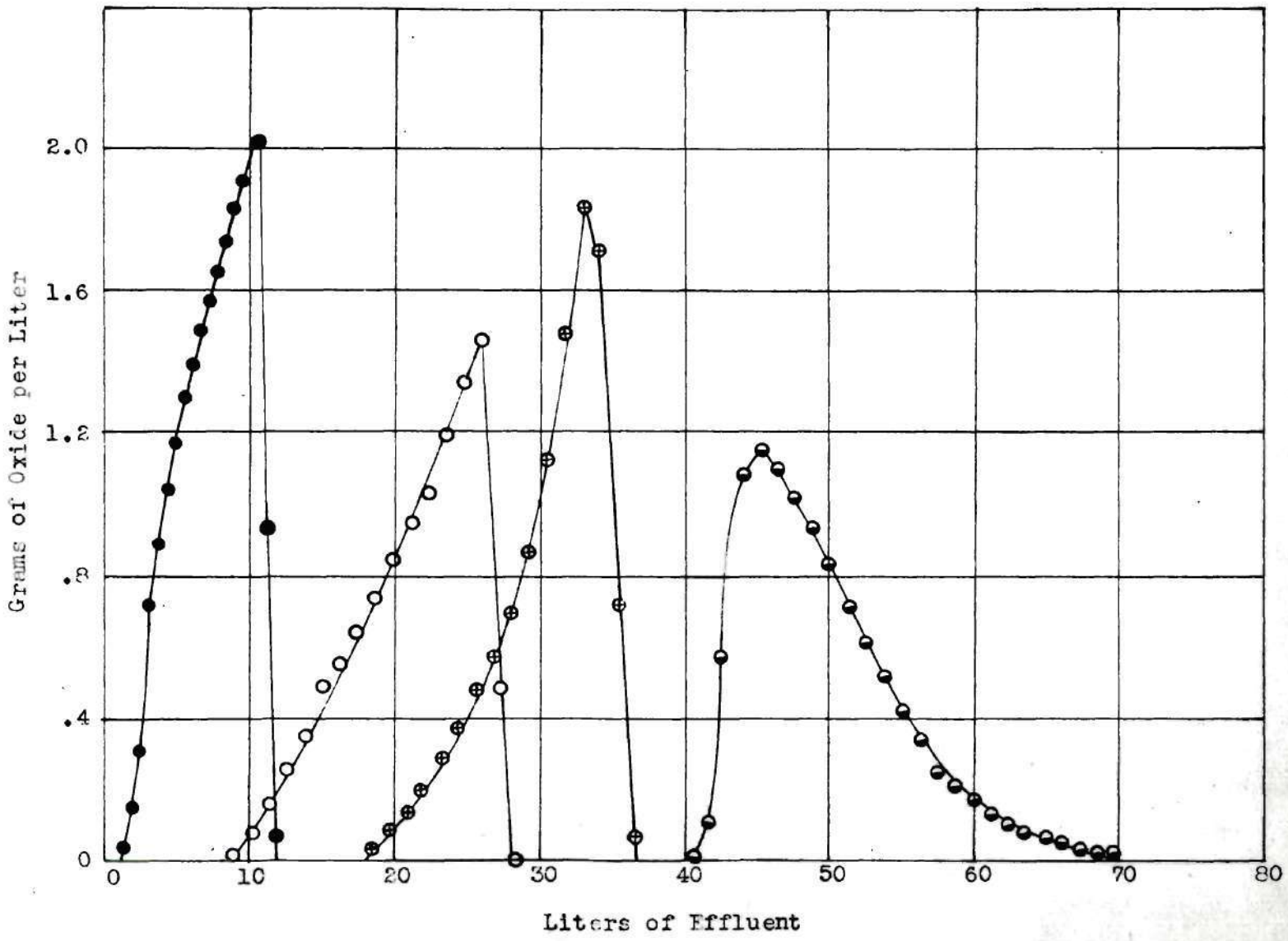
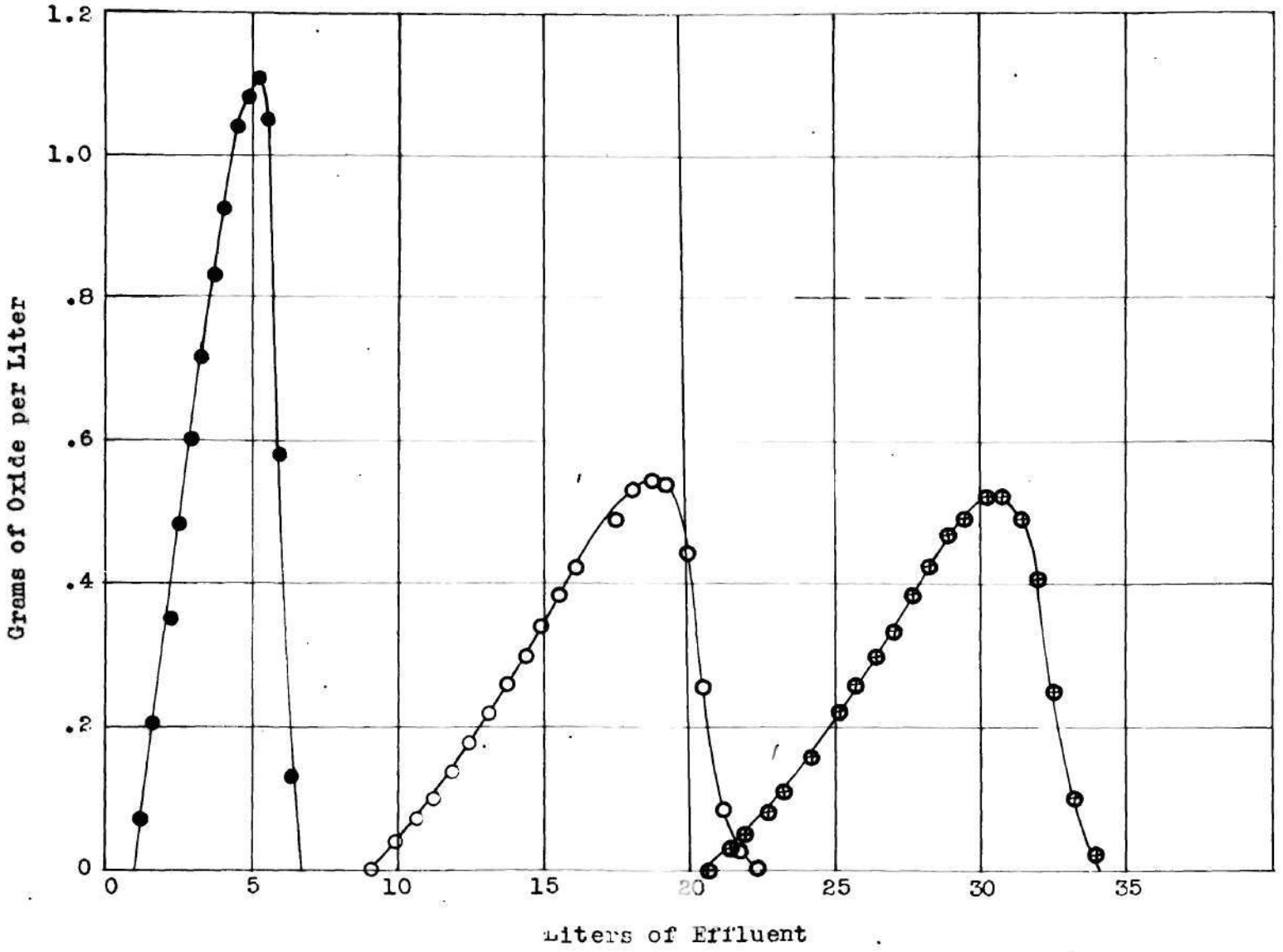

Grams of Oxide per Liter
2.0
1.6
1.2
.8
.4
0
0
10
20
30
40
50
60
70
80
Liters of Effluent

Figure 7

Effect of pH on the Elution of Neodymium for

Column Loading of 3.60 grams of Nd_2O_3

Curve Designation	Run Number	pH of Eluent	Column Loading gm Nd_2O_3	Average Flow Rate ml/min
⊕—⊕—⊕—⊕	46	3.11	3.602	10.0
○—○—○—○	44	3.20	3.602	20.4
●—●—●—●	50	3.50	3.600	19.3


1.2
1.0
.8
.6
.4
.2
0
Grams of Oxide per Liter
0
5
10
15
20
25
30
35
Liters of Effluent

If the theoretical elution curves in Figure 1 are compared with the actual elution curves in Figure 6 and it is assumed that non-equilibrium column operation has caused the sharp edges of the equilibrium curves to become diffuse, the following similarities will be noted: (1) the actual elution curve at pH 3.00 is similar to the theoretical curve A5, (2) the actual elution curves at pH 3.11, 3.20, and 3.50 are similar to the theoretical curve C5. The above analogy is also possible for praseodymium by comparing the theoretical elution curves in Figure 1 with the actual elution curves for praseodymium in Figure 5.

In the development of the theory we assumed a certain adsorption isotherm and predicted the general shape of the elution curve. This procedure can now be reversed and by examining the elution curves it is possible to predict the shape of the adsorption isotherms. If we would now equilibrate neodymium or praseodymium resin with five per cent citric acid solution of various pHs, the adsorption isotherms should have the shape of curve C1 in Figure 1 at pH 3.00, and the shape of curve B1 in Figure 1 at some intermediate pH between 3.00 and 3.12.

On examining the effect of pH of eluent on the peak concentration it is noted that as the pH is increased above 3.12 for neodymium (Figure 6), the peak concentration passes through a minimum. This minimum is not exhibited by the elution curves for praseodymium (Figure 5) and neodymium (Figure 7) at the pHs studied. Further experiments are necessary to determine if this effect is exhibited by praseodymium at some intermediate pH.

A similar minimum for neodymium has been observed in earlier

experiments which were carried out in this laboratory on mixed oxide runs. It has also been exhibited in pure neodymium runs carried out by Spedding[21] on Amberlite IR-100 resin beds with 0.5 per cent citrate solutions.

Effect of Column Loading

The effect of column loading on the elution of pure neodymium at pH 3.20 is illustrated in Figure 8. It is noted from the curves in Figure 8 that as the column loading is increased the position of the peak is moved to the right. It will also be noted that the position of the break-through is inconsistent. It would be expected that increasing the column loading would cause the break-through to occur sooner due to the decrease in the effective length of the column. It is believed that these inconsistencies in the position of the break-through are the result of slight changes in the variables within the limits of experimental error. Thus, examination of Figure 6 shows that an increase of pH from 3.00 to 3.20, a change of 0.20 pH units, has caused the break-through to occur 31.4 liters sooner. Since the pH measurements are only reproducible to $\pm$0.02 pH units, the position of the break-through is only reproducible to about $\pm$3 liters.

Since the area under the elution curve is directly proportional to the weight of rare earth initially adsorbed on the column, this area must increase as the loading is increased. This increase in area is achieved by an increase in the concentration of rare earth at the peak

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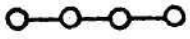
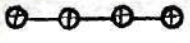
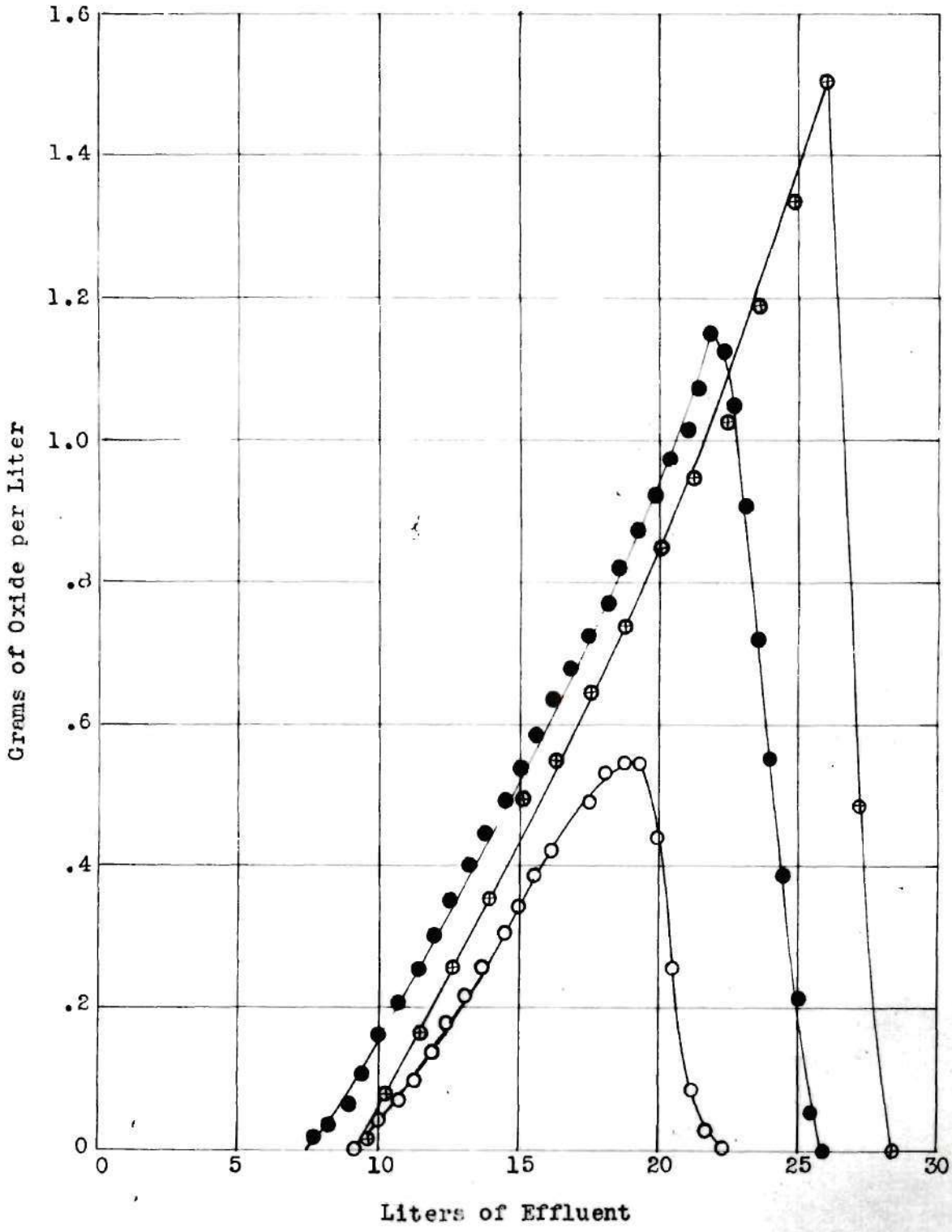
[21] Spedding, F.H., Fulmer, E.I., Butler, T.A., and Powell, J.E., J. Am. Chem. Soc., 72, 2349 (1950).

Figure 8

Effect of Column Loading on the Elution of Neodymium

Curve Designation	Run Number	pH of Eluent	Column Loading gm Nd_2O_3	Average Flow Rate ml/min
	44	3.20	3.602	20.4
	32	3.20	10.000	18.6
	43	3.20	13.024	20.2


1.6
1.4
1.2
1.0
.8
.6
.4
.2
0
0
5
10
15
20
25
30
Grams of Oxide per Liter
Liters of Effluent

and also by an increase in the width of the elution band. Figures 9 and 10 illustrate the effect of column loading on the band width and peak concentration, respectively. It will be noted from Figure 10 that the peak concentration is directly proportional to the column loading over an extensive range at pH 3.20. Further experimental data is needed to establish the exact nature of the curves at pH 3.11 and 3.50.

Effect of Flow Rate

Since the experiments made to determine the effect of pH on the elution process indicated that a change of 0.02 pH units caused a change of about 3 liters in the position of the break-through, an operating procedure was needed to minimize any possible variation in the pH of the eluent between different column runs. To minimize any possible error in the pH of the eluent it was necessary to prepare a supply of eluent which would be sufficiently large to carry out all the flow rate experiments at one column loading. The effect of flow rate on the elution of pure neodymium was studied at two column loadings. Figures 11 and 12 represent the effect of flow rate at column loadings of 13.0 and 3.60 grams of oxide respectively.

Examination of Figure 11 indicates that increasing the flow rate decreases the concentration at the peak, increases the width of the band, and causes break-through to occur sooner. The combined effect is a flattening of the elution band with no appreciable change in the position of the peak.

Similar effects are noted in Figure 12 except with regards to the position of the peak. Increasing the flow rate at the smaller

Figure 9

Effect of Column Loading on Band Width at pH 3.20

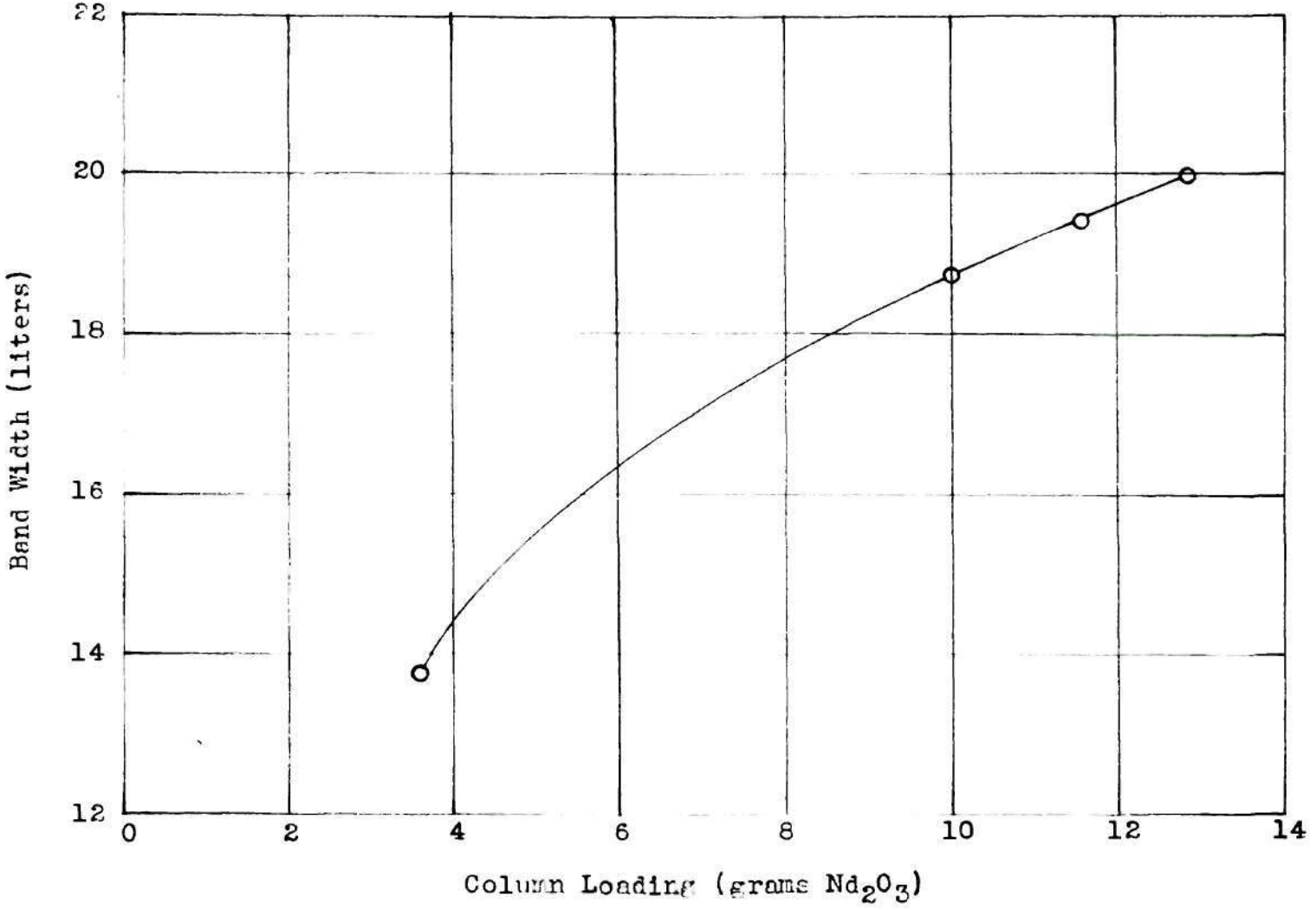

22
20
18
16
14
12
Band Width (liters)
0
2
4
6
8
10
12
14
Column Loading (grams Nd_2O_3)

Figure 10

Effect of Column Loading on Peak Concentration

Curve Designation	pH of Eluent
⊕—⊕—⊕—⊕	3.11
○—○—○—○	3.20
●—●—●—●	3.50

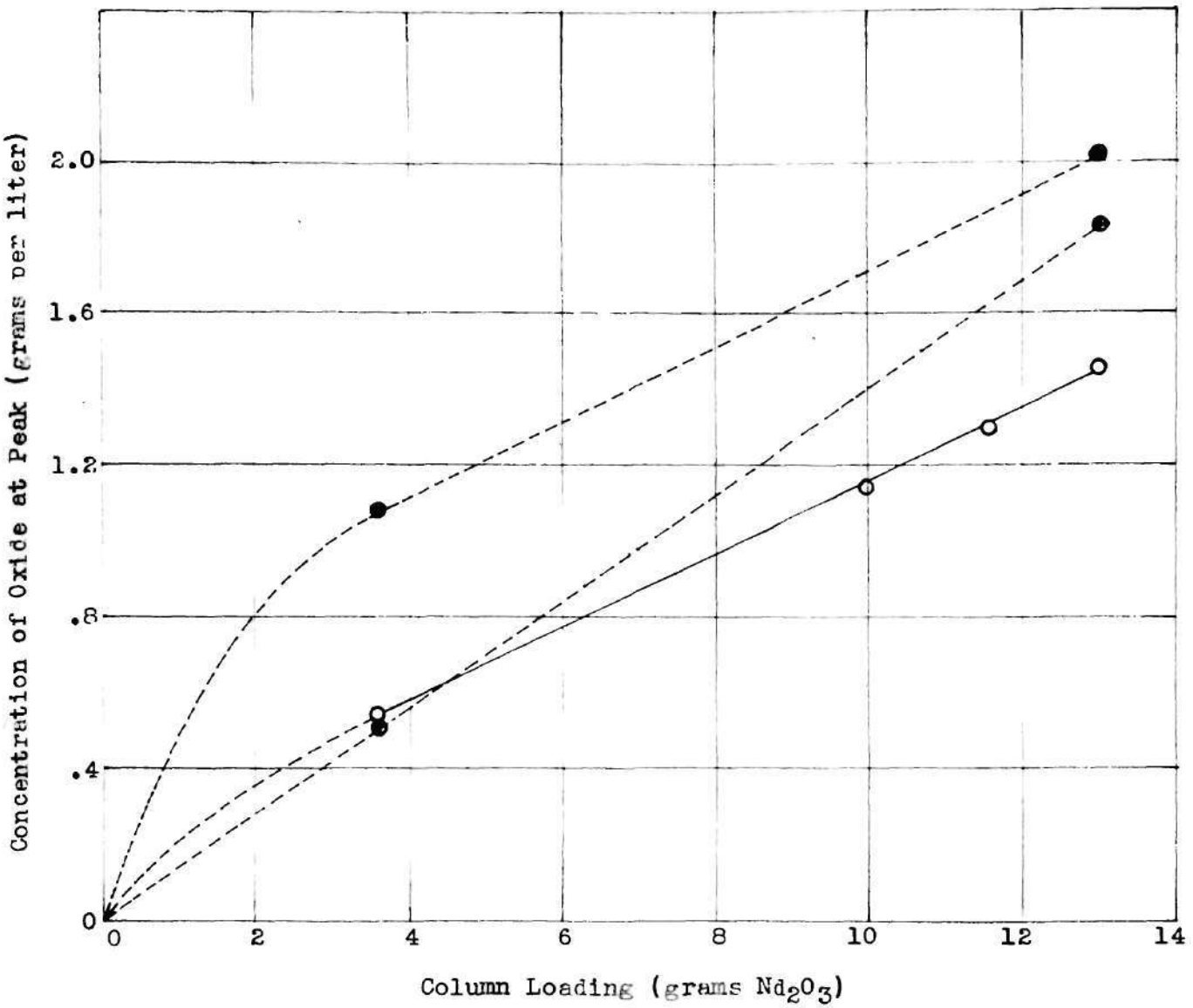

Concentration of Oxide at Peak (grams per liter)
2.0
1.6
1.2
.8
.4
0
0
2
4
6
8
10
12
14
Column Loading (grams Nd_2O_3)

Figure 11

Effect of Flow Rate on the Elution of Neodymium

Curve Designation	Run Number	pH of Eluent	Column Loading gm Nd_2O_3	Average Flow Rate ml/min
●—●—●—●	40	3.11	13.021	10.3
○—○—○—○	45	3.11	13.001	20.0
⊕—⊕—⊕—⊕	41	3.11	13.002	31.3

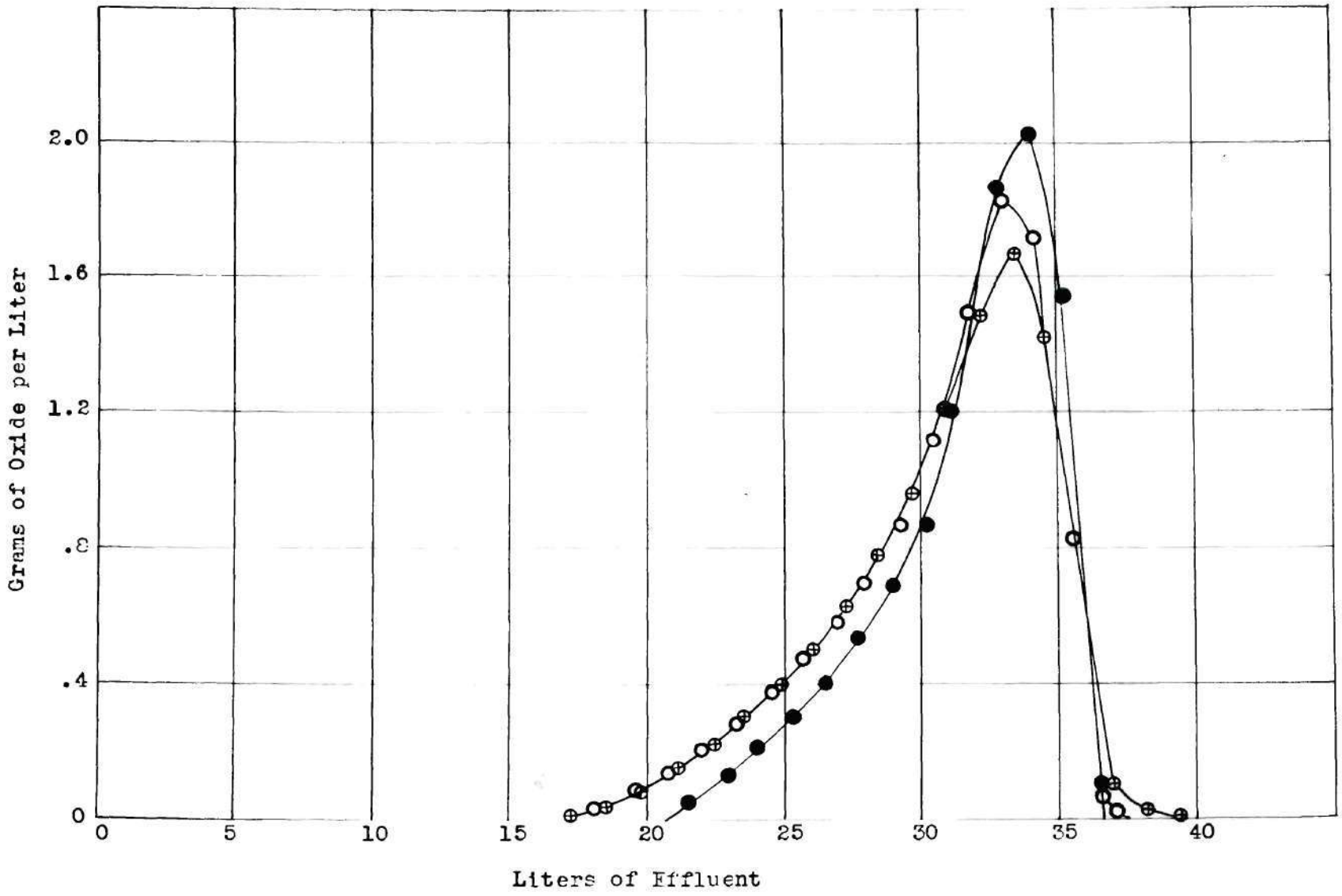
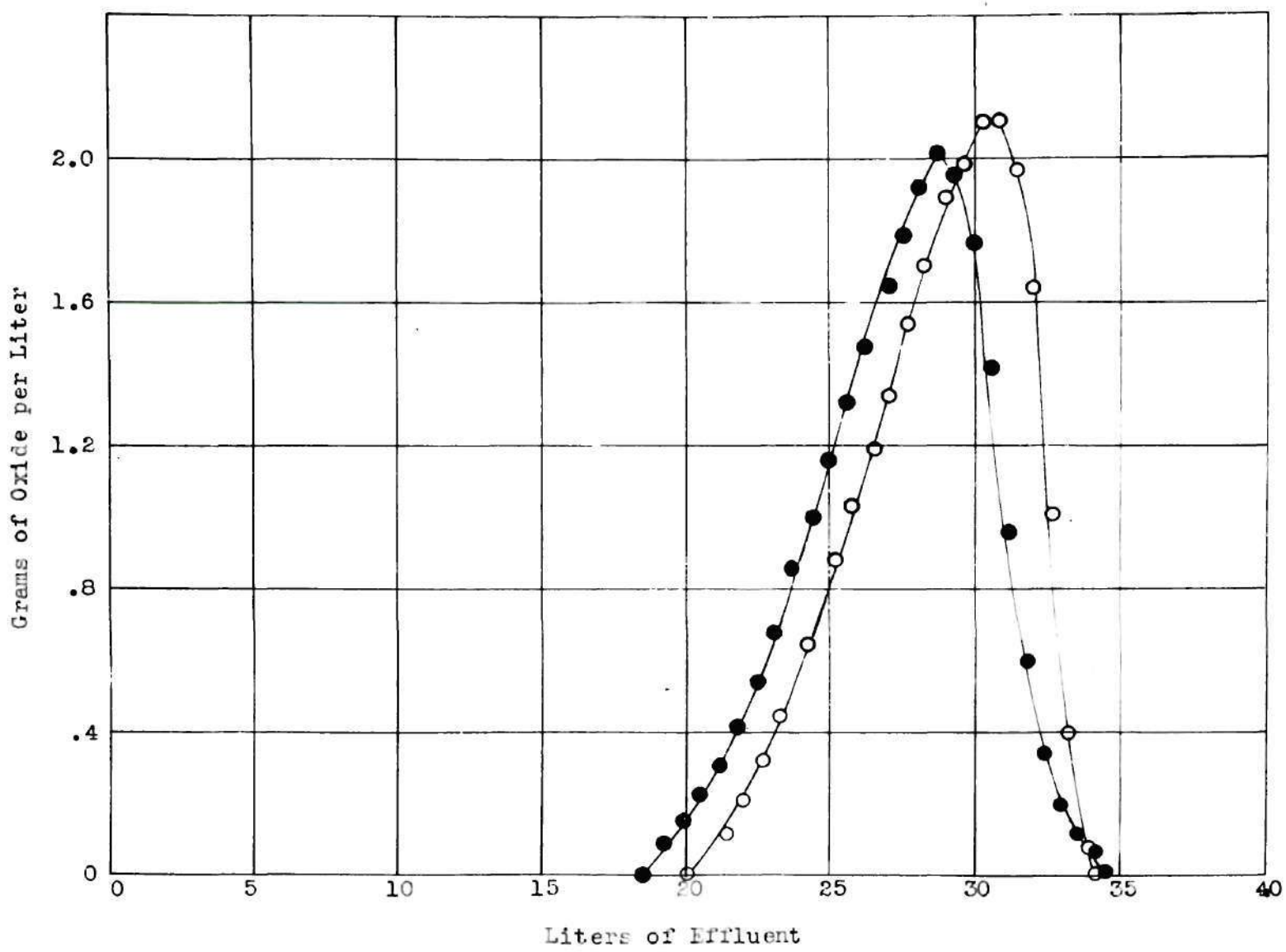

Grams of Oxide per Liter
2.0
1.6
1.2
.8
.4
0
0
5
10
15
20
25
30
35
40
Liters of Effluent

Figure 12

Effect of Flow Rate on the Elution of Neodymium

Curve Designation	Run Number	pH of Eluent	Column Loading gm Nd_2O_3	Average Flow Rate ml/min
	46	3.11	3.602	10.0
	47	3.11	3.602	32.0


Grams of Oxide per Liter
2.0
1.6
1.2
.8
.4
0
0
5
10
15
20
25
30
35
40
Liters of Effluent

column loading has moved the position of the peak to the left.

Flow rate experiments have been made by other investigators but under somewhat different operating conditions than those described in this paper. To compare the results of different investigators the flow rates are calculated on the basis of a unit cross section of column area. The flow rates of approximately 10, 20, and 30 ml/min used in this thesis correspond to flow rates of 0.58, 1.15, and 1.73 cm/min on the basis of a unit of cross section of column area.

Mayer and Tompkins[22] have carried out flow rate experiments on Dowex 50 resin beds using praseodymium-cerium mixtures in trace quantities and five per cent citrate solutions. The flow rates employed by Mayer and Tompkins were 0.026 and 0.23 cm/min. Their results are in agrement with those illustrated in Figure 11.

Spedding, et al.,[23] have made extensive flow rate experiments with pure neodymium and samarium. Their experiments involved the use of Amberlite IR-100 resin beds, 0.5 per cent citrate solutions, and flow rates of 0.5 and 2 cm/min. The column loadings in Spedding's experiments were of the same order of magnitude as the column loadings employed in this thesis. The results of Spedding's flow rate experiments are quite different in one respect from those illustrated in Figures 11 and 12. Increasing the flow rate in Spedding's experiments has

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[22]Mayer, S.W., and Tompkins, E.R., J. Am. Chem. Soc., 69, 2866 (1947).

[23]Spedding, F.H., Fulmer, E.I., Butler, T.A., and Powell, J.E., J. Am. Chem. Soc., 72, 2349 (1950).

caused the elution bands to flatten, but has also shifted the entire elution band to the right which means that a greater volume of eluent was necessary to produce break-through at the higher flow rate.

The problem of band movement under non-equilibrium elution conditions has as yet not received a rigorous mathematical treatment which will explain the two different experimental phenomena mentioned above. Theoretical studies by Boyd, et al.,[24] and by Walter[25] indicate that an increase in flow rate will cause a widening of the elution band. In Chapter II, two possible band shapes were discussed for the conditions in Figure 2, that is, straight line adsorption isotherms. For the case where non-equilibrium has not wiped out the flat region, B, break-through should require less volume of eluent at the greater flow rate. However, for the case where non-equilibrium operation has caused the edges A and C to overlap, it seems quite possible that break-through could require either a smaller or larger volume of eluent at the greater flow rate. Two processes seem to be involved in the latter case, these are: (1) the greater widening of the elution band at the greater flow rate, and (2) a decrease in the amount of solute transferred down the column per unit volume of eluent passing through the column. This decrease in solute transferred results from the decrease in reaction time at the greater flow rate.

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[24]Boyd, G.E., Myers, L.S. Jr., and Adamson, A.W., J. Am. Chem. Soc., 69, 2849 (1947).

[25]Walter, J. E., J. Chem. Phys., 13, 332 (1945).

Further experimental work is necessary to obtain a more thorough understanding of the non-equilibrium elution process.

CHAPTER V

CONCLUSIONS

The pH of the eluent was found to be the most important factor in determining the shape of the elution curves. By comparing the shape of the actual elution curves with the theoretical elution curves it is possible to predict the general shape of the adsorption isotherms. These comparisons indicated that the results could be explained in terms of an adsorption isotherm having a decreasing slope at pH 3.00, an increasing slope in the pH range 3.12-3.50, and a constant slope at some intermediate pH between 3.00 and 3.12.

Increasing the column loading of Nd_2O_3 resulted in an increase in the band width and an increase in the peak concentration. At pH 3.20, the peak concentration was directly proportional to the column loading over the range of column loadings from 3.60 to 13.0 grams of Nd_2O_3.

The flow rate experiments at a column loading of 13.0 grams of Nd_2O_3 indicated that the greater flow rate required less volume of solution to produce break-through, increased the width of the elution band, and decreased the peak concentration with no appreciable change in the position of the peak. Similar results were obtained at a column loading of 3.60 grams of Nd_2O_3, but in the latter case the peak of the elution curve was reached with less volume of eluent at the greater flow rate.

Flow rate experiments undertaken by Spedding[26] indicated that increasing the flow rate increased the volume of eluent necessary to produce break-through, that is, the entire elution band was shifted together with a widening of the band and a lowering of the peak. Spedding's experiments were carried out on Amberlite IR-100 resin beds using 0.5 per cent citrate solutions.

The discrepancy (in the effect of flow rate on the position of break-through) between the results of the flow rate experiments carried out in this thesis and those carried out by Spedding is not readily explained. Further experimental work is necessary to fully understand the non-equilibrium elution process.

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[26] Spedding, F.H., Fulmer, E.I., Butler, T.A., and Powell, J.E., J. Am. Chem. Soc., 72, 2349 (1950).

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Bauman, W.C., and Eichhorn, J., J. Am. Chem. Soc., 69, 2830 (1947).

Blomeke, J.O., Roberts, J.T., Jr., and Ziegler, W.T., Paper presented before Georgia section - American Chemical Society, Meeting in Miniature, November 17, 1949.

Boyd, G.E., Adamson, A.W., and Myers, L.S. Jr., J. Am. Chem. Soc., 69, 2836 (1947).

Boyd, G.E., Myers, L.S. Jr., and Adamson, A.W., J. Am. Chem. Soc., 69, 2849 (1947).

DeVault, D., J. Am. Chem. Soc., 65, 532 (1943).

Johnson, W.M., Master's Thesis in Chemical Engineering, Georgia Institute of Technology, (1950).

Ketelle, B.H., and Boyd, G.E., J. Am. Chem. Soc., 69, 2800 (1947).

Mayer, S.W., and Tompkins, E.R., J. Am. Chem. Soc., 69, 2800 (1947).

Nachod, F.C., Ion Exchange Theory and Application, Academic Press, Inc., New York, N.Y. (1950).

Roberts, J.T., Jr., unpublished work, Georgia Institute of Technology, (1950).

Spedding, F.H., Fulmer, E.I., Butler, T.A., Bladrow, E.M., Gobush, M., Porter, P.E., Powell, J.E., and Wright, J.M., J. Am. Chem. Soc., 69, 2812 (1947).

Spedding, F.H., Fulmer, E.I., Butler, T.A., and Powell, J.E., J. Am. Chem. Soc., 72, 2349 (1950).

Tompkins, E.R., and Mayer, S.W., J. Am. Chem. Soc., 69, 2859 (1947).

Walter, J. E., J. Chem. Phys., 13, 229 (1945).

Walter, J. E., J. Chem. Phys., 13, 332 (1945).

Wilson, J. N., J. Am. Chem. Soc., 62, 1583 (1940).

Ziegler, W.T., Downs, J.B. Jr., Willeford, V.R. Jr., Roberts, J.T. Jr., and Blomeke, J.O., unpublished work, Georgia Institute of Technology, (1948-1949).

APPENDIX I

Summary of Operating Data for Individual Column Runs

TABLES VI through XXI

In the tables on the following pages the symbols after the fraction numbers indicate the operations given below:

* New batch of eluent added to overhead storage carboys

** Column stopped several hours during fraction

*** Started elution with eluent of pE 3.95

The source of the rare earth oxide used in each of the runs is given below. Additional information on these rare earth oxides is presented in Chapter II, page 19.

Nd_2O_3 Research Chemicals Inc.	Pr_6O_{11} Laboratory Stock	Nd_2O_3 Laboratory Stock
40	36	35
41	38	37
44	42	39
45	49	43
46		48
47		
50		

APPENDIX I

TABLE VI

Summary of Operating Data for

Column Run 32, Elution of Neodymium

Column loading	10.000 gm Nd_2O_3 (0.577 gm/cm^2)
pH of adsorbate solution	2.35
Average flow rate	18.6 ml/min (1.1 ml/cm^2 min)
pH of eluent	3.20
Per cent Nd_2O_3 recovered	99.5

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	Weight of Oxide gm per liter	Color of Oxide
A	400	----	No ppt.		
B	280	2.92	"		
1	455	3.48	"		
2	570	3.43	"		
3	570	3.19	"		
4	555	3.18	"		
5	540	3.18	"		
6	540	----	"		
7	620	3.19	"		
8	620	----	"		
9	615	3.20	"		
10	600	----	"		
11	590	3.19	"		
12	595	----	"		
13	595	3.19	"		
14	580	----	0.009	0.016	pale blue
15	570	3.19	0.021	0.036	"
16	575	----	0.038	0.066	"
17**	630	3.19	0.068	0.108	"
18	620	----	0.100	0.161	"
19	620	3.16	0.127	0.205	"
20	620	----	0.157	0.253	"
21	620	3.16	0.187	0.302	"
22	610	----	0.213	0.349	"
23	600	3.16	0.239	0.398	"
24	605	----	0.271	0.447	"
25	610	3.14	0.301	0.493	"
26	600	----	0.323	0.539	"
27	590	3.13	0.345	0.585	"

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TABLE VI (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
28	600	----	0.379	0.632	pale blue
29	600	3.13	0.407	0.679	"
30	590	----	0.428	0.725	"
31	580	3.12	0.448	0.773	"
32	590	----	0.484	0.821	"
33	590	3.11	0.515	0.873	"
34	580	----	0.534	0.921	"
35	570	3.10	0.558	0.978	"
36**	435	----	0.444	1.022	"
37	520	3.10	0.563	1.082	"
38	430	----	0.496	1.153	"
39	450	3.11	0.508	1.129	"
40	460	----	0.485	1.054	pale gray blue
41	460	3.12	0.419	0.910	"
42	455	----	0.329	0.723	"
43	450	3.16	0.250	0.555	"
44	460	----	0.179	0.389	"
45	460	3.19	0.100	0.216	"
46	450	----	0.027	0.060	"
47	565	3.20	no ppt.		

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TABLE VII

Summary of Operating Data for

Column Run 35, Elution of Neodymium

Column loading	12.816 gm Nd_2O_3 (0.739 gm/cm^2)
pH of adsorbate solution	2.20
Average flow rate	16.0 ml/min (0.925 ml/cm^2 min)
pH of eluent	3.12
Per cent Nd_2O_3 recovered	99.4

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	550	3.93	no ppt.		
B	420	2.97	"		
1	1100	3.40	"		
2	1140	3.15	"		
3	1010	3.12	"		
4	880	3.12	"		
5	820	3.12	"		
6	760	3.12	"		
7	720	3.12	"		
8	680	3.12	"		
9	660	3.12	"		
10	630	3.12	"		
11	620	3.12	"		
12	600	3.12	"		
13	580	3.12	"		
14	570	3.12	"		
15	1260	3.12	"		
16	1175	3.12	"		
17	1180	3.12	0.024	0.020	pale blue gray
18*	1140	3.11	0.073	0.064	"
19	1140	3.11	0.143	0.126	"
20	1200	3.11	0.239	0.199	"
21	1170	3.09	0.319	0.273	"
22	1130	----	0.397	0.351	"
23	1130	3.08	0.484	0.429	pale blue
24	1100	----	0.565	0.514	"
25	1100	3.07	0.663	0.603	"
26	1070	----	0.746	0.697	"
27	1070	3.04	0.856	0.800	"

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TABLE VII (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
28	1040	----	0.963	0.926	pale blue
29	1050	3.01	1.116	1.063	"
30	1020	----	1.283	1.258	"
31	1030	2.99	1.536	1.491	pale blue gray
32	1000	----	1.726	1.726	pale blue
33	1020	2.99	1.382	1.355	"
34	990	----	0.228	0.230	"
35	1000	3.10	trace		
36	980	----	no ppt.		

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TABLE VIII

Summary of Operating Data for

Column Run 36, Elution of Praseodymium

Column loading	13.033 gm Pr_6O_{11} (0.752 gm/cm^2)
pH of adsorbate solution	2.12
Average flow rate	19.4 ml/min (1.1 ml/cm^2 min)
pH of eluent	3.12
Per cent Pr_6O_{11} recovered	99.7

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	610	3.40	no ppt.		
B	445	2.98	"		
1	1100	3.41	"		
2	1140	3.10	"		
3	1202	----	"		
4	1150	3.12	"		
5	1140	----	"		
6	1110	3.12	"		
7	1110	----	"		
8	1080	3.12	"		
9	1080	----	"		
10	1060	3.12	"		
11	1070	----	"		
12	1050	3.12	"		
13	1050	----	"		
14	1035	3.12	"		
15	1040	----	"		
16	1020	3.12	"		
17	1030	----	"		
18	1130	3.12	"		
19*	1245	----	"		
20	1255	----	"		
21	1260	----	"		
22	1255	3.12	0.031	0.025	black
23	1285	----	0.117	0.092	dark brown
24	1260	3.12	0.221	0.176	"
25	1260	----	0.367	0.291	"
26	1230	3.08	0.552	0.449	"
27	1220	----	0.791	0.649	Black

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TABLE VIII (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
28	1200	3.04	1.122	0.935	black
29	1210	----	1.387	1.146	"
30	1195	2.96	1.629	1.363	"
31	1210	2.96	2.084	1.722	"
32	1195	----	2.196	1.838	"
33**	1270	3.00	1.916	1.509	"
34	1360	3.09	0.474	0.348	"
35	1200	----	0.082	0.068	dark brown
36	1160	3.12	0.020	0.017	black
37	1240	3.12	no ppt.		

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TABLE IX

Summary of Operating Data for

Column Run 37, Elution of Neodymium

Column loading	13.052 gm Nd_2O_3 (0.753 gm/cm^2)
pH of adsorbate solution	2.25
Average flow rate	20.6 ml/min (1.2 ml/cm^2 min)
pH of eluent	3.00
Per cent Nd_2O_3 recovered	98.9

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	555	3.69	no ppt.		
B	545	3.10	"		
1	1230	3.36	"		
2	1220	3.00	"		
3	1240	3.00	"		
4	1250	3.00	"		
5	1230	3.00	"		
6	1240	3.00	"		
7	1230	3.00	"		
8	1305	3.00	"		
9	1290	3.00	"		
10	1300	3.00	"		
11	1280	3.00	"		
12	1290	3.00	"		
13	1280	3.00	"		
14	1280	3.00	"		
15	1270	3.00	"		
16	1280	3.00	"		
17	1265	3.00	"		
18*	1340	3.00	"		
19	1300	3.00	"		
20	1240	3.00	"		
21	1220	3.00	"		
22	1230	3.00	"		
23	1220	3.00	"		
24	1220	3.00	"		
25	1210	3.00	"		
26	1220	----	"		
27	1205	3.00	"		

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TABLE IX (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
28	1220	----	no ppt.		
29	1200	3.00	"		
30	1220	----	"		
31	1200	3.00	"		
32	1210	----	"		
33	1190	3.00	0.010	0.008	pale blue gray
34	1210	----	0.133	0.110	"
35	1190	2.95	0.684	0.575	pale blue
36	1200	----	1.309	1.091	"
37	1185	2.90	1.369	1.155	"
38	1195	----	1.317	1.102	"
39*	1180	2.90	1.209	1.024	"
40	1240	----	1.158	0.934	"
41	1235	----	1.031	0.835	"
42	1245	2.93	0.897	0.721	"
43	1230	----	0.758	0.616	"
44	1240	2.96	0.638	0.514	"
45	1220	----	0.512	0.420	"
46	1235	2.97	0.419	0.339	"
47	1225	----	0.310	0.253	"
48	1235	2.98	0.259	0.209	"
49	1225	3.00	0.163	0.133	"
50	1225	3.00	0.163	0.133	"
51	1205	----	0.124	0.103	"
52	1220	3.00	0.098	0.080	"
53	1210	----	0.075	0.062	"
54	1225	3.00	0.058	0.047	"
55	1210	----	0.042	0.035	"
56	1220	3.00	0.031	0.025	"
57	1260	----	0.029	0.023	"
58***	1300	----	0.070		"
59	1270	----	no ppt.		

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TABLE X

Summary of Operating Data for

Column Run 38, Elution of Praseodymium

Column loading	13.048 gm Pr_6O_{11} (0.753 gm/cm^2)
pH of adsorbate solution	2.21
Average flow rate	20.0 ml/min (1.2 ml/cm^2 min)
pH of eluent	3.00
Per cent Pr_6O_{11} recovered	99.2

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	515	3.40	no ppt.		
B	495	3.02	"		
1	1220	3.34	"		
2	1235	2.99	"		
3	1235	2.99	"		
4	1240	3.00	"		
5	1220	3.00	"		
6	1200	----	"		
7	1185	3.00	"		
8	1180	----	"		
9	1180	3.00	"		
10	1180	----	"		
11	1175	3.00	"		
12	1180	----	"		
13	1180	3.00	"		
14	1180	----	"		
15	1175	3.00	"		
16	1175	----	"		
17	1180	3.00	"		
18	1180	----	"		
19	1175	3.00	"		
20*	1175	----	"		
21	1340	----	"		
22	1135	3.00	"		
23	1200	----	"		
24	1195	3.00	"		
25	1180	----	"		
26	1175	3.00	"		
27	1170	----	"		
28	1175	3.00	"		

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TABLE X (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
29	1175	----	no ppt.		
30	1175	3.00	"		
31	1175	----	"		
32	1170	3.00	"		
33	1170	----	"		
34	1160	3.00	"		
35	1160	----	"		
36	1160	3.00	"		
37	1160	----	"		
38	1155	3.00	"		
39	1160	----	"		
40*	1160	3.00	"		
41	1240	----	"		
42	1235	3.00	trace		
43	1220	----	0.182	0.150	dark brown
44	1200	2.93	0.539	0.449	"
45	1195	----	0.728	0.609	brown black
46	1200	2.92	0.814	0.678	"
47	1195	----	0.832	0.696	"
48	1190	2.91	0.788	0.663	"
49	1190	----	0.742	0.624	"
50	1190	2.92	0.692	0.581	"
51	1185	----	0.641	0.541	"
52	1185	2.93	0.592	0.499	"
53	1180	----	0.551	0.467	"
54	1180	2.95	0.503	0.426	"
55	1180	----	0.471	0.399	"
56	1180	2.95	0.436	0.370	"
57	1170	----	0.398	0.337	"
58	1175	2.96	0.369	0.314	"
59	1175	----	0.339	0.289	"
60**	1175	2.97	0.312	0.265	"
61	1630	----	0.389	0.238	"
62	820	----	0.173	0.211	"
63	1220	2.97	0.243	0.199	"
64	1220	2.98	0.220	0.180	"
65	1215	----	0.197	0.162	"
66	1240	2.99	0.181	0.146	"
67	1240	----	0.166	0.134	"
68	1245	2.99	0.150	0.121	"
69	1245	----	0.137	0.110	"

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TABLE X (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	Weight of Oxide gm per liter	Color of Oxide
70	1245	----	0.123	0.099	brown black
71	1250	----	0.110	0.088	"
72	1245	2.99	0.100	0.080	"
73	1245	----	0.091	0.073	"
74	1250	----	0.083	0.066	"
75	1240	----	0.074	0.060	"
76	1280	----	0.070	0.054	"
77	1340	3.00	0.069	0.051	"
78	1320	----	0.058	0.044	"
79	1310	----	0.049	0.037	"
80	1200	3.00	0.041	0.034	"
81	1165	----	0.036	0.030	"
82	1175	----	0.032	0.027	"
83***	1570	----	0.207		dark brown
84	1560	----	no ppt.		

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TABLE XI

Summary of Operating Data for

Column Run 40, Elution of Neodymium

Column loading	13.021 gm Nd_2O_3 (0.751 gm/cm^2)
pH of adsorbate solution	2.15
Average flow rate	10.32 ml/min (0.60 ml/cm^2 min)
pH of eluent	3.11
Per cent Nd_2O_3 recovered	97.8

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	Weight of Oxide gm per liter	Color of Oxide
A	495	3.47	no ppt.		
B	505	3.01	"		
1	1230	3.38	"		
2	1220	3.10	"		
3	1210	3.11	"		
4	1170	3.11	"		
5	1220	3.12	"		
6	1270	----	"		
7	1260	3.11	"		
8	1250	----	"		
9	1240	3.11	"		
10	1230	----	"		
11	1220	3.11	"		
12*	1220	----	"		
13	1315	3.11	"		
14	1250	----	"		
15	1240	3.12	"		
16	1240	----	"		
17	1240	3.11	"		
18	1230	----	0.061	0.050	pale blue
19	1230	3.11	0.151	0.123	"
20	1220	----	0.255	0.209	"
21	1220	3.10	0.366	0.301	"
22	1220	----	0.491	0.403	"
23	1220	3.07	0.647	0.530	"
24	1215	----	0.836	0.688	"
25*	1210	3.05	1.060	0.876	"
26	1240	----	1.487	1.199	"
27	1290	2.97	2.413	1.871	"

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TABLE XI (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
28	1290	----	2.860	2.217	pale blue
29	1270	2.98	1.967	1.549	"
30	1270	----	0.135	0.107	"
31	1260	3.11	no ppt.		

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TABLE XII

Summary of Operating Data for

Column Run 41, Elution of Neodymium

Column loading	13.002 gm Nd_2O_3 (0.750 gm/cm^2)
pH of adsorbate solution	2.30
Average flow rate	31.3 ml/min (1.8 ml/cm^2 min)
pH of eluent	3.11
Per cent Nd_2O_3 recovered	99.1

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	Weight of Oxide gm per liter	Color of Oxide
A	500	4.75	no ppt.		
B	500	2.98	"		
1	1230	3.37	"		
2	1250	3.10	"		
3	1260	3.11	"		
4	1240	3.11	"		
5	1270	----	"		
6	1280	----	"		
7	1260	3.11	"		
8	1295	----	"		
9	1300	3.11	"		
10	1275	----	"		
11	1305	----	"		
12	1315	----	"		
13	1285	3.11	"		
14	1315	----	0.013	0.010	pale blue
15	1320	----	0.046	0.035	"
16	1290	----	0.103	0.080	"
17*	1310	3.11	0.188	0.144	"
18	1240	----	0.275	0.221	"
19	1205	----	0.368	0.305	"
20	1240	3.08	0.492	0.397	"
21	1230	----	0.621	0.505	"
22	1200	3.06	0.758	0.631	"
23	1225	----	0.954	0.779	"
24	1230	3.02	1.188	0.966	"
25	1200	----	1.452	1.210	"
26	1220	2.98	1.823	1.494	"
27	1220	----	2.034	1.668	"
28	1200	2.99	1.705	1.421	gray blue

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TABLE XII (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	Weight of Oxide gm per liter	Color of Oxide
29	1220	----	0.722	0.592	gray tan
30	1220	3.08	0.120	0.100	gray brown
31	1190	----	0.027	0.023	light brown
32	1220	3.11	trace		
33	1220	----	no ppt.		

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TABLE XIII

Summary of Operating Data for

Column Run 42, Elution of Praseodymium

Column loading	13.017 gm Pr_6O_{11} (0.751 gm/cm^2)
pH of adsorbate	2.15
Average flow rate	19.4 ml/min (1.1 ml/cm^2 min)
pH of eluent	3.20
Per cent Pr_6O_{11} recovered	99.6

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	510	4.11	no ppt.		
B	490	3.01	"		
1	1095	3.47	"		
2	1245	3.18	"		
3	1180	----	"		
4	1135	3.20	"		
5	1105	----	"		
6	1080	3.20	"		
7	1070	----	"		
8	1055	3.20	"		
9	1050	----	"		
10	1030	3.20	"		
11	1030	----	"		
12	1020	3.20	"		
13**	1160	3.20	0.017	0.015	dark brown
14	1235	----	0.069	0.056	"
15	1225	3.15	0.151	0.124	"
16*	1250	----	0.257	0.205	"
17	1230	3.15	0.361	0.293	"
18	1220	----	0.470	0.385	"
19	1235	----	0.594	0.481	brown black
20	1230	----	0.720	0.585	"
21	1230	----	0.861	0.700	"
22	1220	3.13	1.033	0.847	"
23	1220	----	1.236	1.013	"
24	1210	3.10	1.504	1.243	"
25	1210	----	1.874	1.549	"
26	1200	3.04	2.180	1.816	"
27	1205	----	1.450	1.204	black
28	1200	3.17	0.188	0.157	brown
29	1200	----	no ppt.		

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TABLE XIV

Summary of Operating Data for

Column Run 43, Elution of Neodymium

Column loading	13.024 gm Nd_2O_3 (0.751 gm/cm^2)
pH of adsorbate solution	2.01
Average flow rate	20.2 ml/min (1.2 ml/cm^2 min)
pH of eluent	3.20
Per cent Nd_2O_3 recovered	98.9

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	475	4.28	no ppt.		
B	495	3.10	"		
1	1170	3.39	"		
2	1210	3.18	"		
3	1240	3.20	"		
4	1235	3.20	"		
5	1210	----	"		
6	1210	3.20	"		
7	1210	----	"		
8	1205	3.20	0.020	0.017	pale blue
9	1210	----	0.095	0.079	"
10	1200	3.19	0.197	0.164	"
11	1210	----	0.310	0.256	"
12	1200	3.17	0.418	0.348	"
13	1200	----	0.592	0.493	"
14	1200	3.14	0.657	0.547	"
15	1200	----	0.776	0.647	"
16	1200	3.13	0.885	0.738	"
17**	1260	----	1.065	0.845	"
18	1215	3.11	1.153	0.949	"
19	1220	----	1.257	1.030	"
20	1220	3.10	1.456	1.193	"
21	1220	----	1.642	1.346	"
22	1210	3.07	1.771	1.464	"
23	1210	----	0.589	0.487	tan
24	1210	3.20	trace		
25	1210	----	no ppt.		

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TABLE XV

Summary of Operating Data for

Column Run 44, Elution of Neodymium

Column loading	3.602 gm Nd_2O_3 (0.208 gm/cm^2)
pH of adsorbate solution	2.30
Average flow rate	20.4 ml/min (1.2 ml/cm^2 min)
pH of eluent	3.20
Per cent Nd_2O_3 recovery	98.8

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	Weight of Oxide gm per liter	Color of Oxide
A	310	4.14	no ppt.		
B	505	3.56	"		
1	1100	3.50	"		
2	1240	3.21	"		
3	1240	3.20	"		
4	1195	----	"		
5	1200	3.20	"		
6	1175	----	"		
7	1180	3.21	"		
8**	605	----	"		
9	670	----	trace		
10	1060	3.20	0.042	0.040	pale blue
11	295	----	0.021	0.071	"
12	660	----	0.065	0.100	tan
13	615	3.20	0.085	0.138	pale blue
14	620	----	0.111	0.179	"
15	620	----	0.136	0.219	"
16	610	3.19	0.157	0.258	"
17	600	----	0.181	0.301	"
18	615	----	0.209	0.340	"
19	615	3.18	0.237	0.385	"
20	605	----	0.255	0.422	"
21	680	----	0.278	0.409	"
22	615	3.18	0.302	0.491	"
23	615	----	0.327	0.531	"
24	605	----	0.331	0.546	"
25	600	3.17	0.325	0.542	"
26	620	----	0.273	0.440	"
27	610	----	0.155	0.254	gray blue

APPENDIX I

TABLE XV (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
28	595	3.19	0.051	0.086	tan
29	600	----	0.016	0.027	"
30	610	----	trace		
31	610	----	no ppt.		

APPENDIX I

TABLE XVI

Summary of Operating Data for

Column Run 45, Elution of Neodymium

Colum loading	13.001 gm Nd_2O_3 (0.750 gm/cm^2)
pH of adsorbate solution	2.55
Average flow rate	20.0 ml/min (1.2 ml/cm^2 min)
pH of eluent	3.11
Per cent Nd_2O_3 recovered	99.5

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	505	3.67	no ppt.		
B	470	3.07	"		
1	1180	3.39	"		
2	1250	3.11	"		
3	1290	3.11	"		
4	1220	3.11	"		
5	1240	----	"		
6	1120	----	"		
7	1195	----	"		
8	1175	----	"		
9	1190	----	"		
10	1175	----	"		
11	1200	----	"		
12	1180	----	"		
13	1195	3.12	"		
14	1175	----	"		
15	1190	3.11	"		
16	1170	----	0.033	0.028	pale blue
17	1190	3.11	0.095	0.080	"
18	1170	----	0.157	0.134	"
19	1190	3.10	0.239	0.201	"
20	1170	----	0.331	0.283	"
21	1180	3.08	0.443	0.375	"
22*	1210	----	0.576	0.476	"
23	1230	----	0.708	0.576	"
24	1200	3.04	0.838	0.698	"
25	1220	----	1.058	0.868	"
26	1205	3.02	1.352	1.122	"
27	1210	----	1.808	1.494	"

APPENDIX I

TABLE XVI (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	Weight of Oxide gm per liter	Color of Oxide
28	1210	2.90	2.222	1.836	pale blue
29	1230	----	2.111	1.716	"
30	1220	3.05	0.883	0.724	"
31	1220	----	0.076	0.062	pale blue gray
32	1200	3.11	no ppt.		

APPENDIX I

TABLE XVII

Summary of Operating Data for

Column Run 46, Elution of Neodymium

Column loading	3.602 gm Nd_2O_3 (0.208 gm/cm^2)
pH of adsorbate solution	2.20
Average flow rate	10.0 ml/min (0.577 ml/cm^2 min)
pH of eluent	3.11
Per cent Nd_2O_3 recovered	99.4

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	350	3.44	no ppt.		
B	502	3.13	"		
1	1130	3.11	"		
2	1035	3.11	"		
3	1020	----	"		
4	1000	----	"		
5	1140	----	"		
6	1300	----	"		
7	1180	----	"		
8	1230	----	"		
9	1220	----	"		
10	598	----	"		
11	1200	----	"		
12	597	----	"		
13	597	----	"		
14	595	----	"		
15	600	----	"		
16	590	3.10	"		
17	605	----	"		
18	590	----	"		
19	600	3.09	"		
20	590	----	"		
21	600	----	"		
22	595	3.08	"		
23	605	----	"		
24	595	----	"		
25	600	----	trace		
26	595	----	trace		
27*	750	----	0.021	0.029	pale blue

APPENDIX I

TABLE XVII (Cont.)

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
28	630	----	0.033	0.052	pale blue
29	620	----	0.051	0.081	"
30	630	----	0.065	0.111	"
31					
32	1250	----	0.183	0.162	"
33	633	----	0.139	0.220	"
34	625	----	0.161	0.258	"
35	630	----	0.187	0.297	"
36	613	----	0.208	0.334	"
37	625	----	0.241	0.385	"
38	615	----	0.262	0.427	"
39	620	----	0.292	0.472	"
40	622	----	0.309	0.497	"
41	620	----	0.326	0.526	"
42	610	----	0.321	0.525	"
43	620	----	0.305	0.492	"
44	620	----	0.254	0.409	"
45	620	----	0.156	0.252	"
46	610	----	0.052	0.101	"
47	622	----	0.013	0.021	"
48	315	----	no ppt.		

APPENDIX I

TABLE XVIII

Summary of Operating Data for

Column Run 47, Elution of Neodymium

Column loading	3.602 gm Nd_2O_3 (0.208 gm/cm^2)
pH of adsorbate solution	2.20
Average flow rate	32.0 ml/min (1.8 gm/cm^2 min)
pH of eluent	3.11
Per cent Nd_2O_3 recovered	99.3

Fraction Number	Volume ml	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	325	no ppt.		
B	320	"		
1	1235	"		
2	1275	"		
3	1300	"		
4	1305	"		
5	1310	"		
6	1320	"		
7	1305	"		
8	1315	"		
9**	1350	"		
10	675	"		
11	665	"		
12	650	"		
13	655	"		
14	660	"		
15	660	"		
17	650	"		
18	640	"		
19	640	"		
20	650	trace		
21	645	0.014	0.022	pale blue
22	650	0.025	0.038	"
23	645	0.036	0.055	"
24	620	0.047	0.077	"
25	630	0.065	0.104	"
26	635	0.086	0.136	"
27*	665	0.113	0.170	"
28	635	0.135	0.213	"

APPENDIX I

TABLE XVIII (Cont.)

Fraction Number	Volume ml	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
29	630	0.157	0.250	pale blue
30	610	0.177	0.291	"
31	620	0.204	0.329	"
32	630	0.232	0.368	"
33	625	0.257	0.411	"
34	630	0.282	0.447	"
35	620	0.299	0.482	"
36	600	0.301	0.502	"
37	620	0.303	0.489	"
38	610	0.271	0.444	"
39	620	0.219	0.354	"
40	620	0.150	0.242	"
41	610	0.092	0.150	"
42	605	0.052	0.086	gray blue
43	605	0.030	0.050	"
44	620	0.018	0.030	"
45	615	0.011	0.018	"
46	610	no ppt.		

APPENDIX I

TABLE XIX

Summary of Operating Data for

Column Run 48, Elution of Neodymium

Column loading	13.161 gm Nd_2O_3 (0.759 gm/cm^2)
pH of adsorbate solution	2.20
Average flow rate	19.4 ml/min (1.1 ml/cm^2 min)
pH of eluent	3.50
Per cent Nd_2O_3 recovered	99.3

Fraction Number	Volume ml	Mean pH	Weight of Oxide gm per fraction	Weight of Oxide gm per liter	Color of Oxide
A	500	4.92	no ppt.		
B	400	3.00	"		
1	670	----	"		
2	230	----	"		
3	530	----	0.022	0.041	pale blue gray
4	600	----	0.150	0.251	pale blue
5	600	----	0.306	0.509	"
6	600	----	0.432	0.719	"
7	580	3.44	0.518	0.893	"
8	590	----	0.614	1.041	"
9	590	----	0.696	1.179	"
10	585	3.40	0.764	1.306	"
11	570	----	0.796	1.397	"
12	575	----	0.858	1.493	"
13	580	3.38	0.909	1.568	"
14	580	----	0.955	1.647	"
15	560	----	0.973	1.737	"
16	585	3.33	1.072	1.832	"
17	585	----	1.121	1.917	"
18	580	3.31	1.162	2.003	"
19	565	3.32	1.143	2.022	"
20	575	3.40	0.539	0.937	gray tan
21	575	3.50	0.039	0.067	pale gray blue
22	580	----	no ppt.		

APPENDIX I

TABLE XX

Summary of Operating Data for

Column Run 49, Elution of Praseodymium

Column loading	12.994 gm Pr_6O_{11} (0.750 gm/cm^2)
pH of adsorbate solution	2.20
Average flow rate	18.9 ml/min (1.1 ml/cm^2 min)
pH of eluent	3.50
Per cent Pr_6O_{11} recovered	99.5

Fraction Number	Volume ml	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	510	no ppt.		
B	500	"		
1	540	"		
2	552	"		
3	622	"		
4	558	0.025	0.045	brown black
5	600	0.113	0.189	dark brown
6	600	0.220	0.366	"
7	590	0.314	0.533	"
8	580	0.392	0.677	brown black
9	578	0.466	0.805	"
10	580	0.535	0.922	"
11	576	0.592	1.028	"
12	558	0.629	1.127	"
13	565	0.685	1.212	"
14	570	0.737	1.293	"
15	565	0.776	1.373	"
16	550	0.785	1.427	"
17	560	0.852	1.521	"
18	560	0.883	1.576	"
19	560	0.908	1.621	"
20	555	0.951	1.714	"
21	550	1.001	1.821	"
22	555	1.048	1.889	"
23	555	0.861	1.551	"
24	540	0.154	0.285	"
25	550	trace		
26	555	no ppt.		

APPENDIX I

TABLE XXI

Summary of Operating Data for

Column Run 50, Elution of Neodymium

Column loading	3.600 gm Nd_2O_3 (0.208 gm/cm^2)
pH of adsorbate solution	2.47
Average flow rate	19.3 ml/min (1.1 ml/cm^2 min)
pH of eluent	3.50
Per cent Nd_2O_3 recovered	98.6

Fraction Number	Volume ml	Weight of Oxide gm per fraction	gm per liter	Color of Oxide
A	525	no ppt.		
B	500	"		
1	368	"		
2	336	"		
3	410	"		
4	380	0.027	0.070	pale blue gray
5	405	0.083	0.206	"
6	406	0.143	0.352	pale blue
7	400	0.194	0.484	"
8	400	0.240	0.601	"
9	405	0.290	0.717	"
10	400	0.332	0.831	"
11	390	0.361	0.926	"
12	380	0.382	1.004	"
13	375	0.405	1.081	"
14	380	0.421	1.108	"
15	381	0.401	1.053	"
16	380	0.222	0.585	"
17	380	0.050	0.131	"
18	373	trace		
19	395	no ppt.		